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FINDINGS OF THE U.S. DEPARTMENT OF DEFENSE TECHNOLOGY ASSESSMENT TEAM ON JAPANESE HIGH-TEMPERATURE COMPOSITES FEBRUARY 1989 VISIT

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13. ABSTRACT *(Maximum 200 words)*

In January 1989 a technology assessment team on high-temperature composites, led by Dr. Benjamin A. Wilcox of ARPA's Materials Sciences Division, conducted a three-week visit to Japanese industry. The purpose of the TAT visit to Japan was threefold: (1) to assess Japanese technology in high-temperature structural composites and reinforcing fibers in order to identify opportunities for improving the performance and durability of U.S. defense hardware; (2) to communicate the observations to U.S. government and industry; and (3) to foster cooperation/coordination in the translation and coupling of such technology in the production of U.S. defense hardware. Technical details of interest included: precursor materials; processing techniques and equipment; availability, applications, cost and performance of fibers and composites; and standards and procedures for nondestructive inspections. Particular emphasis was focused on present and future applications, manufacturing procedures, and production capability. In Section II a report of each visit to a Japanese company is provided, with full technical detail. In Section III the technical information is reviewed and discussed by subject matter. In Section IV, various technical and nontechnical findings are summarized. A discussion of the thermal stability of fibers and composites and environmental effects on them appears in Appendix F.

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INSTITUTE FOR DEFENSE ANALYSES

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PREFACE

This report has been prepared for the Advanced Research Projects Agency under the task order entitled "Study of Material Science Areas."

The report was written and edited during 1990. In December of 1990, in accordance with prior agreement, the report was forwarded to the Japan Defense Agency and the Ministry of International Trade and Industry for their comments or concurrence. Periodic inquiries during late 1991 and 1992 via the Mutual Defense Assistance Office in the American Embassy in Tokyo failed to elicit any response. Hence the report is being published as written.

ACKNOWLEDGMENTS

The Technology Assessment Team (TAT) on High-Temperature Composites gratefully acknowledges the assistance of many people in the United States and Japan. Col. Robert C. Johnson, Director, Far East/Middle East Affairs in the Pentagon [and formerly Chief of the Mutual Defense Assistance Office, Japan (MDO) in the American Embassy in Tokyo] made the original arrangements with MDO and traveled to Japan to facilitate the first contacts with the Japan Defense Agency (JDA) and the Ministry of International Trade and Industry (MITI). He also served as the primary contact in the Pentagon for the TAT throughout the planning, visiting, and reporting cycle.

Captain Walter T. Dziedzic, Chief of MDO, his deputy chief, Norman S. Hastings, and Raymond Y. Aka, Interpreter, were of great assistance in Japan, especially during the TAT visits to JDA and MITI. Dr. George Wright, Director, Science Liaison Office Far East, and his successor, Dr. Arthur F. Findeis, provided various administrative support and lent three of their staff to serve as members of the TAT.

The TAT is deeply indebted to many people in JDA and MITI who arranged and supported the access to Japanese industry. Among them are Teruo Suzuki, Director General for Research and Development, Ryozo Tsutsui, Director General of the Technical Research and Development Institute, and Katsuro Shinzeki and Yoshifumi Fujita, Director and Deputy Director, Coordination Division, Equipment Bureau, all of JDA, and Hidehiro Konno, Director of Aircraft and Ordnance Division, and his successor, Shinichiro Ohta, both of MITI. The TAT also thanks Akira Ryuzaki, Motoi Satake, and Shigeyoshi Hata, all of JDA, who took turns escorting the TAT in its visits to Japanese industry.

The TAT is most particularly indebted to Jamieson C. Allen, Director of R&D Exchange in MDO. His insights, contacts, and assistance in arrangements with JDA and MITI and throughout the visits to Japanese industry were invaluable.

Lastly, the TAT acknowledges the hospitality and thanks all the Japanese companies and their staffs who made the visit of the TAT so pleasant and technically rewarding.

CONTENTS

GLOSSARY

ABS	Acrylonitrile-Butadiene-Styrene
ACC-J	American Chamber of Commerce-Japan
ArFRP	Aramid Fiber-Reinforced Plastic
BMI	Bismaleimide
CAD	Computer-Aided Design
CAE	Computer-Aided Engineering
CAM	Computer-Aided Manufacturing
CASTEM	Casting Analysis System (KOBELCO)
C-C	Carbon-Carbon (Composite)
CERASEP	SiC-SiC CMC Made by SEP
CF	Carbon Fiber
CFRP	Carbon-Fiber-Reinforced Plastic
CIP	Cold Isostatic Press
CIPNAS	CIP Numerical Analyis System (KOBELCO)
CMC	Ceramic Matrix Composite
COMPGLAS	A Family of Fiber-Reinforced Glass and Glass Ceramic CMC
CRDL	Corporate Research and Development Laboratory
CRP	Carbon-Reinforced Plastic
CTE	Coefficient of Thermal Expansion
CVD	Chemical Vapor Deposition
CVI	Chemical Vapor Infiltration
DARPA	Defense Advanced Research Projects Agency
DoD	Department of Defense
EP	Epoxy
ERS	Earth Resource Satellite

FCC	Fluid Catalytic Cracking
FRC	Fiber-Reinforced Ceramic
FRM	Fiber-Reinforced Metal
FRP	Fiber-Reinforced Plastic
FSX	U.S.-Japan Jointly Produced Fighter Aircraft
FW	Filament-Wound
GEO	Geostationary Earth Orbit
GFRP	Graphite-Fiber-Reinforced Plastic
HIP	Hot Isostatic Press
HIPNAS	HIP Numerical Analysis System (KOBELCO)
HM	High Modulus
HT	High Temperature; High Tensile Strength
HVR	High-Volume Resistivity
I&IP	Office of the Deputy Under Secretary of Defense for Industrial and International Programs
IHI	Ishikawajima-harima Heavy Industries Company, Ltd.
IHPTET	Integrated High-Performance Turbine Engine Technology
IP&T	Office of the Deputy Under Secretary of Defense for International Programs and Technology
ISAS	Institute of Space and Aeronautical Science (Japan)
JASDF	Japan Air Self-Defense Force
JDA	Japan Defense Agency
JGDF	Japan Ground Self-Defense Forces
JRDC	Japan Research Development Corporation
K.K.	Kabushiki Kaisha (Joint Stock Company)
KOBELCO	Kobe Steel, Ltd.
KSI	Kips (Thousands of Pounds) per Square Inch
LAS	Lithium Aluminosilicate
L.C.	Liquid Crystal
LVR	Low-Volume Resistivity

M/C-C	Metal/Carbon-Carbon
MDA	Mutual Defense Assistance
MDO	Mutual Defense Assistance Office, Japan
MELCO	Mitsubishi Electric Company
MITI	Ministry of International Trade and Industry (Japan)
MMC	Metal Matrix Composite
MSI	Mips (Millions of Pounds) per Square Inch
NAL	National Aerospace Laboratory (Japan)
NASA	National Aeronautics and Space Administration
NASDA	National Space Development Agency (Japan)
NASP	National Aerospace Plane
NCK	Nippon Carbon Company, Ltd.
NDE	Nondestructive Evaluation
NTPQ	A Complex Polymer (Fig. 17)
ODS	Oxide Dispersion Strengthened
PAN	Polyacrylonitrile
PBT	Polybutylene Terephthalate
PEEK	Polyetherketone
PET	Polyethylene Terephthalate
PMC	Plastic Matrix Composite
P-N	Positive-Negative
PPQ	Polyphenylquinoxaline
PPS	Polypara-phenylene Sulfide
PSZ	Partially Stabilized Zirconia
PVA	Polyvinyl Alcohol
R&AT	Office of the Deputy Under Secretary of Defense for Research and Advanced Technology
R&D	Research and Development
RSR	Rapid Solidification Rate
RT	Room Temperature

SEM	Scanning Electron Microscope
SEP	Société Européenne de Propulsion
Sialon	Ceramics Based on Si-Al-O-N Chemistry
TAT	Technology Assessment Team
TC	Temperature Coefficient
Tonen	Toa Nenryo Kogyo Co., Ltd.
TP	Thermoplastic
TRDI	Technical Research and Development Institute, JDA
TS	Thermoset
TSK	Tonen Sekiyu Kagaku K.K.
UHM	Ultrahigh Modulus
U.K.	United Kingdom
UTRC	United Technologies Research Center
UV	Ultraviolet
3D	Three-Dimensional

EXECUTIVE SUMMARY

A. BACKGROUND

In September 1980 the Japan-U.S. Systems and Technology Forum was established as an informal medium for meetings between senior working-level defense officials in the United States and Japan to promote cooperation in the fields of equipment and technology. In January 1983 Japan agreed to open the way for the transfer of military technologies to the United States. The Office of the Deputy Under Secretary of Defense for International Programs and Technology (IP&T) was responsible for implementing this new agreement with Japan via the Systems and Technology Forum. IP&T had the assistance, in Japan, of the Mutual Defense Assistance Office (MDO) in the American Embassy. In the United States, IP&T had the assistance of the Office of the Deputy Under Secretary of Defense for Research and Advanced Technology (R&AT). R&AT was responsible for assembling teams of U.S. scientists to identify areas of interest, to visit appropriate Japanese facilities, and to assess the potential for mutual benefits from the transfer of technology.

B. INTRODUCTION AND PURPOSE

In late January 1989, Dr. Ben A. Wilcox, of the Materials Sciences Division of the Defense Advanced Research Projects Agency (DARPA), led a Technology Assessment Team (TAT) on High-Temperature Composites on a visit to Japanese industry. The team members are identified in Table S-1, and the industries visited and their activities of interest are shown in Table S-2.

The purpose of the TAT visit to Japan was to assess Japanese technology in high-temperature structural composites and reinforcing fibers in order to identify opportunities for improving the performance and durability of U.S. defense hardware; to communicate the observations to U.S. government and industry; and to foster cooperation/coordination in the translation and coupling of such technology in the production of U.S. defense hardware. Technical details of interest included: precursor materials; processing

Table S-1. Team Members, High-Temperature Composites TAT

Dr. Ben A. Wilcox, Team Leader, Assistant Director for Materials Sciences, Defense Advanced Research Projects Agency (DARPA), Arlington, Virginia.
Mr. Jamieson C. Allen, Director, Defense Technology Trade Programs, Mutual Defense Assistance Office, U.S. Embassy, Tokyo, Japan.
Mr. Charles F. Bersch, Science and Technology Division, Institute for Defense Analyses (IDA), Alexandria, Virginia.
Dr. Steven G. Fishman, Program Manager, Office of Naval Research (ONR), Arlington, Virginia.
Dr. Shiro Fujishiro, Associate Director, Air Force Office of Scientific Research--Far East (AFOSR-FE), Tokyo, Japan.
Dr. Allan P. Katz, Materials Research Engineer, Air Force Wright Research and Development Center (WRDC)--Materials Laboratory, Wright-Patterson Air Force Base (AFB), Ohio.
Dr. Ed Lenoe, Assistant Director, Army Research Office--Far East (ARO-FE), Tokyo, Japan.
Dr. Merrill L. Minges, Director, Nonmetallic Materials Division, Air Force Wright Aeronautical Laboratories (AFWAL)--Materials Laboratory, Wright-Patterson AFB, Ohio.
Dr. Fred Pettit, Assistant Director, Office of Naval Research--Far East (ONR-FE), Tokyo, Japan.

Table S-2. Fiber and Composite Activities of Japanese Companies Visited*

Company	Continuous Fibers (f) or Whiskers (w)		Composites			
	Ceramic	Graphite	Metal Matrix	Ceramic Matrix	Carbon-Carbon	Organic Matrix
Ube Industries (visited 1/31/89)	● f,w		o	●		
Kobe Steel (visited 2/1/89)	● w	● f	o	o	●	o
Shimadzu Sanjo Works (visited 2/2/89)	● f					
Toray Industries (visited 2/3/89)		● f	o			●
Toyota Corporate Research and Development Laboratory (visited 2/6/89)			●	o		
IHI (visited 2/7/89)			o	o		●
Mitsubishi Electric Corp. (visited 2/8/89)			o	o		●
Tonen -- General Labs (visited 2/9/89) -- Kawasaki Works (visited 2/13/89)	● f	● f		o		o
Nippon Carbon (visited 2/10/89)	● f	o f	●	●	●	

● = strong activity; o = lesser activity.

* The TAT also visited Keidanren (Japan Federation of Economic Organizations) on 2/14/89, making a presentation to more than 100 members of Keidanren on three areas: (1) composites of interest to the U.S. Department of Defense (DoD); (2) composites of interest for the National Aerospace Plane (NASP); and (3) composites of interest for Integrated High-Performance Turbine Engine Technology (IHPTET).

techniques and equipment; availability, applications, cost and performance of fibers and composites; and standards and procedures for nondestructive inspection. Particular emphasis was to be focused on present and future applications, manufacturing procedures, and production capability.

C. GENERAL FINDINGS

The general findings of the TAT may be summarized as follows:

- The influence of the Japan Defense Agency (JDA) and the Ministry of International Trade and Industry (MITI) helped a great deal in opening doors for the TAT. The 9 Japanese companies (at 10 installations) were, by and large, very open in their discussions and plant tours. In some cases the access to and flow of information was unprecedented.
- Most U.S. R&D in fibers and composites has been for high-performance, often high-temperature, aerospace applications. Most composite research and some fiber research have been supported by the Department of Defense (DoD) and the National Aeronautics and Space Administration (NASA). Any commercial applications followed the Government-sponsored developments. In contrast, Japanese R&D in fibers and composites has largely been company sponsored. Japanese companies have been laying the groundwork--positioning themselves in sporting goods, and preparing for future markets in automotive and, eventually, aerospace applications. Their efforts may be high technology but are not high temperature at present. The use of both Nicalon and Kevlar fibers in the bow of a tennis racket and graphite fibers in the handle illustrates the high-technology interest of the Japanese consumer.
- Japanese materials scientists and engineers are very capable and well trained, very industrious, and very persevering. The Japanese have about 30,000 young scientists and engineers engaged in advanced materials R&D. They often pursue a technology where no payoff is apparent; they believe an application can be found for a good material. The costs of development and time--up to 15 years--are sometimes enormous. Japanese patience and perseverance is impressive.
- Some attrition in materials programs was apparent: (1) At one time about 30 Japanese companies were working on carbon fibers; there are many less now. Toray dropped pitch fibers to focus on PAN fibers. Tonen emphasizes pitch. Nippon Carbon transferred its share of a joint PAN effort to Asahi but continues to produce pitch-based carbon fibers. (2) Several companies have stopped R&D of particulate/whisker ceramic matrix composites (CMCs) in favor of monolithic ceramics; they claim processing costs outweigh any likely improvements in performance.

- Many Japanese companies seek licensing and distribution relationships with U.S. companies: Nippon Carbon distributes Nicalon fibers through Dow Corning, and Ube Industries distributes Tyranno fibers via Textron Specialty Materials, while Toray has cross-licenses on some high-performance resins with Hexcel.
- Japanese pilot plants are BIG: one fiber processing line was at least 250 feet long. The plants are able to wring out the potential of the equipment and the process in a realistic manufacturing setting as well as to find potential problems. In contrast, DoD-supported pilot plants are often "tabletop glassware" operations.
- The Japanese closely follow worldwide developments in composites and other technologies. For example, Keidanren (Japan Federation of Economic Organizations) collected DARPA's Annual Report to Congress for the years 1979-1987, combined and restructured it by technology subject, translated it, and distributed it to Japanese industry.

D. TECHNICAL FINDINGS

The technical findings of the TAT may be summarized as follows:

- Japan is very strong in carbon/graphite fibers. Toray, with PAN-based fibers, and Tonen, with pitch-based fibers, are outstanding. Current emphases are on cost reduction and improvements in resin matrices and fiber-matrix interfaces in order to maximize translation of the fiber properties into the composite.
- Japan appears to lead the world in high-performance ceramic fibers prepared from organosilicon polymeric precursors: Nippon Carbon has Nicalon; Ube Industries has Tyranno; and Tonen seems about ready to market a silicon nitride fiber.
- Fiber production operations are well designed and constructed (e.g., several spinning and curing operations are done in clean rooms). Most operations are highly automated, with minimal manpower required.
- Some preceramic polymers appear to have great potential for additional product applications such as oxidation protection coatings, tapes, films, adhesives, and laminated macrocomposites. Tailored "hybrid" reinforcement schemes seem likely to evolve.
- The TAT encountered considerable activity in metal matrix composites (MMCs), but mostly with aluminum as the matrix and short fibers as the reinforcement. Squeeze casting is well developed and seems to be the preferred fabrication technique. Toyota is producing MMC in piston heads for

diesel engines, but the TAT saw no indication of commodity MMC forms such as Dural in the United States is said to produce.

- The TAT encountered almost no activity in high-temperature MMCs, that is, almost nothing in titanium or intermetallic matrices. However, this observation requires two qualifications. First, the TAT did not visit Fuji, Kawasaki, or Mitsubishi Heavy Industries, all known to be active in all types of composites. Second, just before the TAT arrived in Japan, the Japanese Diet approved a new MITI Advanced Composite Materials Program ($80 million from the government over 8 years; additional funds from participating industry are sometimes 10 times greater than the government money) to start in April 1989, with emphasis on high-temperature composite applications for aerospace and the Japanese spaceplane, HOPE, which is to be designed in 1992. Virtually all of the companies the TAT visited expressed great enthusiasm for their respective roles in the program.

- The interest and activity of the Japanese in continuous-fiber CMCs appears to have been limited because they saw no market. Promising efforts, such as Ube's Tyrannohex, Nippon Carbon's Nicaloceram and LAS materials, and Tonen's Si_3N_4 matrix composites, were all in early stage of development at modest effort levels. This is likely to change with the new MITI initiative described above.

- Japanese interest in particulate/whisker-reinforced CMCs seemed to be waning. Several companies said that processing costs were prohibitive and that the mechanical properties of monolithic ceramics were comparable to those of discontinuously reinforced ceramics. The TAT saw several instances of monolithic ceramics, especially silicon nitride, being used in automotive turbocharger rotors.

- Just as in the United States, there is a very poor data base on the performance of ceramic and graphite fibers in MMCs and CMCs. There is a particular need for more information on the present capability and thermal stability of ceramic fibers at high temperatures.

- The Japanese are accelerating their research in C-C, especially in the new MITI program. Besides C-C work observed by the TAT at Kobe Steel and Nippon Carbon, it was reported that Mitsubishi Heavy Industries and the Space Division of Nissan Motors produce C-C components for rocket engines. Expanded applications in aircraft brakes are expected soon. The TAT saw no signs of work for gas-turbine-engine or reentry applications. However, promising developments in fibers and mesophase pitch suggest rapid growth in the MITI program.

I. BACKGROUND, INTRODUCTION, PURPOSE, AND REPORT STRUCTURE

A. BACKGROUND

In 1954 the United States and Japan completed the Mutual Defense Assistance (MDA) Agreement, under which U.S. equipment and technologies were provided to Japan to improve the Japanese self-defense capability. In September 1980 the Japan-U.S. Systems and Technology Forum was established as an informal medium for meetings between senior working-level defense officials in the United States and Japan to promote cooperation in the fields of equipment and technology. In 1981 Dr. Richard D. DeLauer, the Under Secretary of Defense for Research and Engineering, recognized the growing maturity in Japan of many technologies useful to the mutual defense of the United States and Japan and suggested an exchange of defense-related technologies between the two countries.

At that time the Three Principles on Arms Export and the Government Policy Guidelines on Arms Control of February 1976 forbade the export of military technologies from Japan. As a result of Dr. DeLauer's suggestion, the Japanese government studied the relationship between the Three Principles and the Japan-U.S. security arrangements and, in January 1983, agreed to open the way for the transfer of military technologies to the United States. An Exchange of Notes based on the MDA Agreement was concluded in November 1983, and Detailed Arrangements were concluded in December 1985, based on the Exchange of Notes. [Additional details can be found in Appendix A and in *Defense of Japan*, a large (350+ pages) white paper published annually in English by the Japan Defense Agency (JDA) and the Japan Times, Ltd.]

In the United States, the Office of the Deputy Under Secretary of Defense for International Programs and Technology (IP&T)--now the Office of the Deputy Under Secretary of Defense for Industrial and International Programs (I&IP)--was responsible for implementing this new agreement with Japan, via the Systems and Technology Forum. IP&T had the assistance, in Japan, of the Mutual Defense Assistance Office (MDO) in the American Embassy. In the United States, IP&T had the assistance of the Office of the

Deputy Under Secretary of Defense for Research and Advanced Technology (R&AT). R&AT was responsible for assembling teams of U.S. scientists, representing the Department of Defense (DoD), to identify areas of interest, to visit appropriate Japanese facilities, and to assess the potential for mutual benefits from the transfer of technology.

The first Technology Assessment Team (TAT), led by Dr. John M. MacCallum, of the Office of the Deputy Under Secretary of Defense for Research and Engineering/ Research and Advanced Technology/Military Systems Technology (OUSDRE/R&AT/ MST), visited Japan in July 1984 and again in August 1986 to review Japanese programs in electro-optics (EO) and millimeter-wave (MMW) technologies.

The second TAT, led by Dr. Clinton W. Kelly III, then Special Assistant for Strategic Computing at the Defense Advanced Research Projects Agency (DARPA), visited Japan in January 1987 and again in January 1988 to assess Japanese management methods and manufacturing technologies in a wide range of industries.

Reports on the visits of both of those TATs have been published.[1 2]

B. INTRODUCTION AND PURPOSE

In late January 1989, Dr. Ben A. Wilcox, of the Materials Sciences Division of DARPA, led a TAT on High-Temperature Composites on a 3-week visit to Japanese industry. The names and affiliations of the members of the TAT are presented in Appendix B. The schedule of the TAT's activities, including its visits to Japanese government offices and industrial plants and laboratories, is given in Appendix C; this schedule was the product of a brief visit by Dr. Wilcox and Mr. Charles F. Bersch in May 1988 to the Japan Defense Agency (JDA) and the Ministry of International Trade and Industry (MITI) and subsequent negotiations between the MDO of the American Embassy and MITI and JDA.

The purpose of the TAT visit to Japan was threefold: (1) to assess Japanese technology in high-temperature structural composites and reinforcing fibers in order to identify opportunities for improving the performance and durability of U.S. defense hardware; (2) to communicate the observations to U.S. government and industry; and

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1 J.M. MacCallum, Jr., *Electro-Optics and Millimeter-Wave Technology in Japan, Final Report of DoD Technology Team*, Office of the Under Secretary of Defense (Acquisition), Research and Advanced Tehnology, May 1987.

2 C.W. Kelly, III, and J.L. Nevins, *Findings of the U.S. Department of Defense Technology Assessment Team on Japanese Manufacturing Technology*, Charles Stark Draper Laboratory, Inc., Report CSDL-R-2161, June 1989.

(3) to foster cooperation/coordination in the translation and coupling of such technology in the production of U.S. defense hardware. Technical details of interest included: precursor materials; processing techniques and equipment; availability, applications, cost and performance of fibers and composites; and standards and procedures for nondestructive inspection. Particular emphasis was to be focused on present and future applications, manufacturing procedures, and production capability. (Organic polymer matrix composites were excluded by virtue of the emphasis on high-temperature performance.)

When the purpose of the TAT visit was discussed with MITI and JDA in May 1988, it was agreed that the TAT would visit Japanese industry to assess real manufacturing and production capability. This was in contrast to most U.S. scientific visits, which are usually made to laboratories to learn about the status of research. Also at that time MITI suggested that, given the details of interest described above, it might be helpful to submit written questions well before the visits in order to allow time to assemble the information requested and obtain organizational approval for release of the information. The questions forwarded by the TAT prior to the visits appear in Appendix D. The value of the MITI suggestion was reflected by the remarkable openness and hospitality the TAT experienced during its visits.

As a gesture of appreciation for the hospitality shown it, the TAT gave a Thank You Reception at the New Sanno Hotel on the evening of 13 February 1989. The list of guests is presented in Appendix E.

C. STRUCTURE OF THE REPORT

The results of the TAT visits to Japanese industry (observations, discussions, and conclusions) are presented in three ways. In Section II a report of each visit to a Japanese company is provided, with full technical detail. In Section III the technical information is reviewed and discussed by subject matter, but in summary form. In Section IV, various technical and nontechnical findings are summarized. A discussion of the thermal stability of fibers and composites and environmental effects on them appears in Appendix F.

II. VISITS TO INDUSTRY

This section contains reports on all of the TAT visits to Japanese industry, plus a report on a meeting with Keidanren, the Japan Federation of Economic Organizations. Table 1 provides a summary of all the visits and includes a display of the relative activities at each company.

The reader will find that the nature of the visit reports varies considerably. The reason for this is as follows. The TAT wanted to assess the status of advanced fibers and composites in production and, accordingly, on the basis of its best knowledge, had provided a tentative list of proposed places to visit to MITI (via MDO), with a request to upgrade the list in accordance with the TAT's objectives. In the end, the visit list contained only five materials suppliers plus some materials users and R&D laboratories. Fuji, Kawasaki, and Mitsubishi Heavy Industries were removed from the list by MITI because of the then-current political sensitivities surrounding the FSX question. At least one materials supplier requested to be excused, presumably for the same reason. As a consequence, while there was much of interest to the TAT at some stops, at others the amount of information available and pertinent to this report was less.

Further, the reader will find that presentation charts and company data sheets have been copied extensively, and the text has been used to tie the copied material together with the oral comments that accompanied the printed material. The reader is assured that the selection process was rigorous and that the format and the materials selected were chosen to provide the maximum amount of information, precisely as it was received, to a wide variety of readers. Section III provides some further discussions, comparisons, etc.

At least in some instances, the amount of information made available and the scope and extent of facility tours were limited by logistics. The TAT intentionally selected an agenda of one visit per day to maximize information transmission. Even so, because of transportation limitations--from city to city at least once each day outside Tokyo, and from 1 to 2.5 hours traveling time per one-way trip within Tokyo--most visits were less than 6 hours in duration, including lunch.

Table 1. Fiber and Composite Activities of Japanese Companies Visited*

Company	Continuous Fibers (f) or Whiskers (w)		Composites			
	Ceramic	Graphite	Metal Matrix	Ceramic Matrix	Carbon-Carbon	Organic Matrix
Ube Industries (visited 1/31/89)	● f,w		○	●		
Kobe Steel (visited 2/1/89)	● w	● f	○	○	●	○
Shimadsu Sanjo Works (visited 2/2/89)	● f					
Toray Industries (visited 2/3/89)		● f	○			●
Toyota Corporate Research and Development Laboratory (visited 2/6/89)			●	○		
IHI (visited 2/7/89)			○	○		●
Mitsubishi Electric Corp. (visited 2/8/89)			○	○		●
Tonen -- General Labs (visited 2/9/89) -- Kawasaki Works (visited 2/13/89)	● f	● f		○		○
Nippon Carbon (visited 2/10/89)	● f	○ f	○	●	●	

● = strong activity; ○ = lesser activity.

* The TAT also visited Keidanren (Japan Federation of Economic Organizations) on 2/14/89, making a presentation to more than 100 members of Keidanren on three areas of concern: (1) composites of interest to the U.S. Department of Defense (DoD); (2) composites of interest for the National Aerospace Plane (NASP); and (3) composites of interest for Integrated High-Performance Turbine Engine Technology (IHPTET).

The following reports should convey some sense of the extraordinary hospitality and openness with which the TAT was received. Undoubtedly, the official status of the TAT and the TAT acceptance of the MITI suggestion to submit questions beforehand (Appendix D) gave the hosts a sense of the TAT's interests and time to gather information and get release clearances. In nearly every instance the TAT was received at and allowed to enter an industrial facility and to tour pertinent parts. Naturally, the degree of openness varied, but it ranged from unusual to extraordinary.

A. UBE INDUSTRIES, LTD.

The TAT visited the Ube Laboratory of Ube Industries, Ltd. in Ube City in the prefecture of Yamaguchi on 31 January 1989. After the welcome and introduction to Ube Industries, summarized below, there were presentations on silicon nitride powders and whiskers, Tyranno fibers, metal matrix composites, and Tyranno reinforced ceramic matrix composites, and visits to the silicon nitride production plant, the Tyranno fiber pilot plant, and the Ube Scientific Analysis Laboratory, Ltd.

1. Background Information

Ube Industries began as a coal mining company in 1897. Ube Industries, Ltd. was formed in 1942, combining early elements of the current Coal, Chemical, Cement, and Machinery and Plant Engineering Divisions. Today Ube Industries, Ltd. is an international conglomerate of more than 100 companies, including foundries and plants for coal gasification, shipbuilding, industrial machinery, and concrete prefabricated houses. In 1987 total capitalization was ¥42.7 billion and total sales reached ¥469 billion. There were 8300 employees.

In recent years, "high technology" has been emphasized, and to the above four divisions one can now add the Petrochemical, Engineering Plastics, New Products, and Corporate R&D Divisions. The last has laboratories at Ube, Chiba, and Hirokota; they do corporate R&D and interact with the R&D departments of the five primary business divisions. Current R&D (Fig. 1) focuses on electronic materials, engineering ceramics, speciality plastics, and medicines and agrochemicals.

The TAT special interest was in the engineering ceramics category, most particularly the Tyranno fiber development and production and its utilization in metal and ceramic matrix composites.

Ube is doing considerable biotechnology work, but that is not discussed here. At virtually every company the TAT visited, biotechnology was mentioned as a company thrust, but Ube appeared to emphasize it more than the others.

2. Silicon Nitride Powder and Whiskers

Ube produces silicon nitride powder by the decomposition of silicon diimide. They said that high-purity, ultrafine particles and a large amount of α phase aid the sintering (at 1750°C for 4 hours in N_2) of UBE SN-E10 (89.5 percent Si_3N_4, 5 percent Y_2O_3, and

Ever-Expanding Possibilities of Ube's New Materials

Ube, a comprehensive manufacturer of materials, has been making extensive use of diverse technologies. This has resulted in a reservoir of expertise that spurs on the development of new materials capable of responding to new requirements.

Ube's activities encompass a wide range of fields ranging from speciality polymers and engineering ceramics through electronic materials to medicine and agrochemicals. The company's R & D activities which encompass numerous technological fields ranging from organic/inorganic synthesis to biotechnology, have consistently produced outstanding results.

The immense potential of Ube's new materials hold even greater promise for the future.

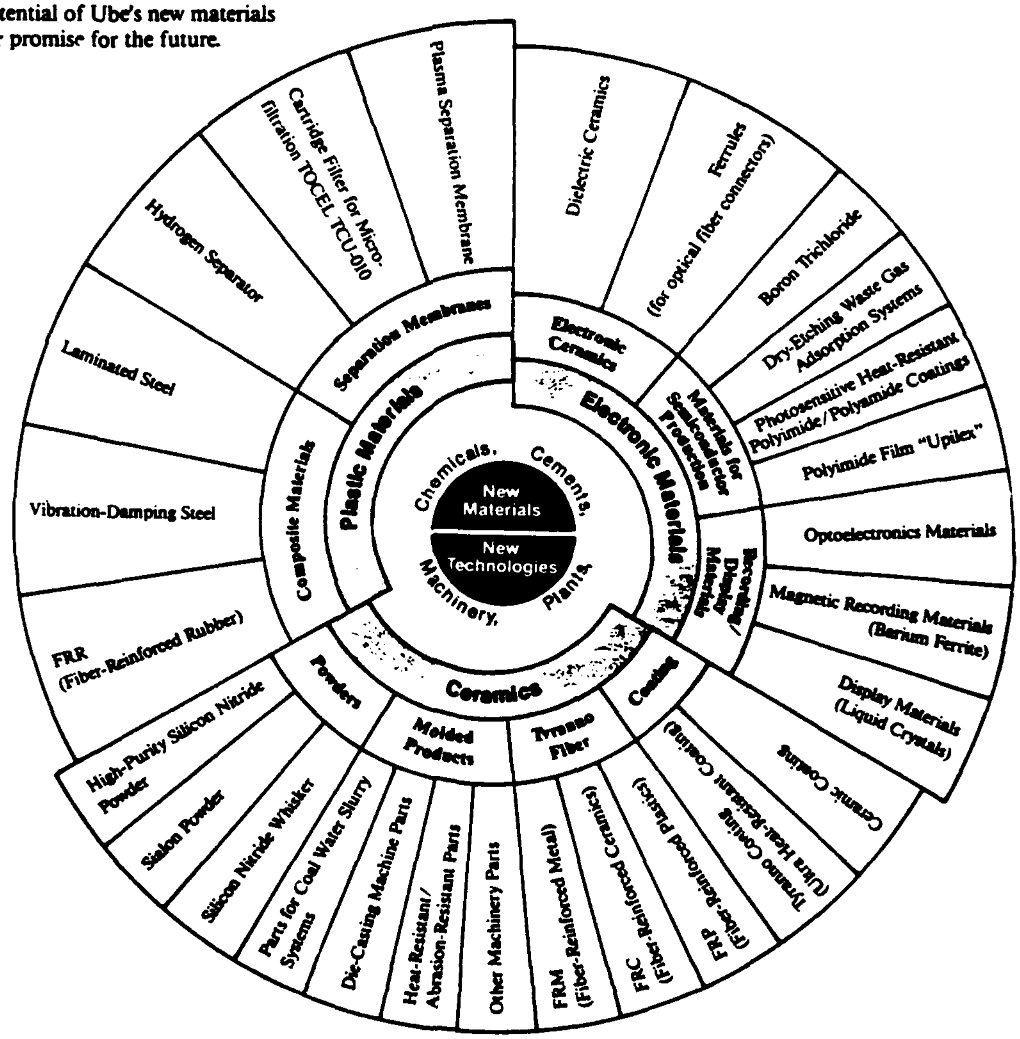


Figure 1

5.5 percent Al_2O_3) to 99 percent of theoretical density (Tables 2, 3). Properties include a flexural strength of greater than 100 kg/mm^2 from room temperature to about 1000°C, a ΔT thermal shock resistance above 900°C, and a K_{IC} of 7.8 MN/m$^{3/2}$. Production capacity is over 100 tons/year, and production rate exceeds 60 tons/year. Ube has made and used dozens of molded and sintered parts in house, including cylinder liners, piston heads, turbine blades, wire drawing dies, rollers, and pulleys--applications where strength at high temperature, corrosion and abrasion resistance, and thermal shock resistance are important. Ube's superfine Si_3N_4 particulate has been widely used by Japanese bearing manufacturers for making Si_3N_4-based ball bearings for machinery applications. It is known in Japan that Ishikawajima-harima Heavy Industries Company, Ltd. (IHI), the F100 engine production licensee for the Japanese F-15, is conducting a serious evaluation of such bearings for potential use in small engines and has produced some optimistic reports.

Ube also uses an imide decomposition process to produce α-type and β-type silicon nitride whiskers (Tables 4, 5) for use in plastic, metal, or ceramic composites, as high-temperature thermal insulation materials, and in plasma spray coatings. The α whiskers are 0.1 to 0.4 microns in diameter and 5-20 microns long, with resulting aspect ratios of 20 to 100. Impurities include 4000-5000 ppm Fe, less than 100 ppm of Al and of Ca, and 2-3 percent O. The β whiskers have diameters of 0.1 to 0.5 microns, lengths of 10-30 microns, and aspect ratios of 20 to 50. Ube claims the large-capacity powder plant enables economical mass production and assures a stable supply.

Ube has used α-type whiskers to reinforce aluminum and β-type whiskers to double the fracture toughness of β-Sialon. Ube has found that the fracture toughness of both α- and β-Sialons increased, to 4.5 and 5.5 MN/m$^{3/2}$, respectively, with a 30 percent addition of whiskers.

3. Tyranno Fibers

Tyranno fibers (Table 6) are amorphous and continuous ceramic fibers of 8.5 or 10.5 micron diameter made by melt-spinning polytitanocarbosilane [polydimethylsilane, tetraalkyl titanium, and a trace of $B(OH)_3$] at 600 meters/minute. Curing on a reel at 200°C for about 6 hours in air and then pyrolyzing in N_2 gas at about 1300°C produces a fiber containing, by weight, 50 percent Si, 29 percent C, 18 percent 0, 2 percent Ti, and less than 0.1 percent B. (Ube says the O is necessary to crosslink the polymer, to improve flexibility and handling, and the Ti, which can be increased up to 7 weight percent, to retain the amorphous character.) The fiber spinning and curing are carried out in a clean room.

Table 2

UBE CERAMICS DEPARTMENT

SILICON NITRIDE POWDER

(LIQUID-INTERFACIAL REACTION METHOD)

UBE INDUSTRIES, LTD. ARK Mori Building 12-32, Akasaka 1-chome, Minato-ku, Tokyo, 107 Japan Phone (03)505-9461 Telex No. 2224645

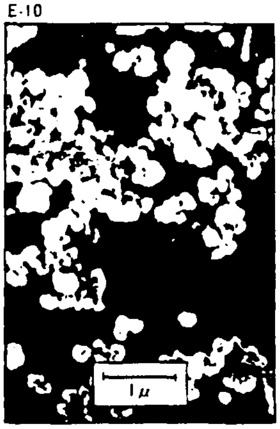
The silicon nitride powder SN-E10, which is synthesized by the liquid-interfacial reaction method developed by UBE INDUSTRIES is of high purity containing a minimum amount of metalic impurities and sub-micron equiaxed particles of high α-phase. Its particle size and morphology are all so uniform and homogeneous that it is most ideal as a high quality raw matherial to be used for production of fine ceramics

SN-E10 has the best sinterability and properties of sintered body
SN-E05 and SN-E02 with more molding easiness are also commercially available

POWDER SPECIFICATION

GRADE	SN-E 10	SN-E 02	SN-E 05
MORPHOLOGY	EQUIAXED	EQUIAXED	EQUIAXED
PARTICLE SIZE	~0.2μm	~1μm	~0.4μm
SPECIFIC SURFACE AREA	10m²/g	2m²/g	5m²/g
PURITY	N >38.0% O <2.0% C <0.2% Cl <100 ppm Fe <100 ppm Ca < 50 ppm Al < 50 ppm		
PHASE COMPOSITION	DEGREE OF CRYSTALLINITY ~ 100% α PHASE > 95% β PHASE < 5%		

E-10



E-02

E-05

LOCATION OF OFFICES

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Phone (03)505-9461 Facsimile (03)505-9234 Telex UBE TK J24318

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Phone (212)765-5865~7 Telex 126187

Dusseldorf Office: Friedrich-Ebert-Strasse 1.4000 Dusseldorf, West Germany
Phone (0211)356085 Telex 8587237

Sydney Office: 1F, Citicorp House, 54 62 Carrington St., Sydney, N.S.W. 2000, Australia
Phone (02)290-2541 Telex 74203

Table 3

Standards values of UBE sintered silicon nitride

	Materials	HIP Si_3N_4	Atmospheric Sintered Si_3N_4
Density	(g/cc)	3.30	3.25
Flexural Strength	(MPa)		
	25℃	1,180	980
	1,000℃	880	880
	1,200℃	590	590
	(kgf/mm²)		
	25℃	120	100
	1,000℃	90	90
	1,200℃	60	60
Toughness Kic	($MN/m^{3/2}$)	5.5	5.5
Vickers Hardness	(kg/mm²)	1,800	1,700
Young's Modulus	($\times 10^4 kg/mm^2$)	3.2	2.8
Poisson's Ratio		0.26	0.26
Thermal Conductivity	(W/mk)	22	12
	(cal/cm·sec.℃)	0.07	0.04
Specific Heat	(J/kg°K)	750	750
	(cal/g℃)	0.18	0.18
Thermal Expansion Coeff	($\times 10^{-6}$/℃)	3.6	3.6
Heat Shock Resistance	(ΔT℃)	>900	>900
Electrical Resistivity	(ohm·cm)	$>10^{14}$	$>10^{14}$

Product design information

- Curvature at the edges or corners of the product should be as great as possible without adversely affecting the function.
- The product's thickness should be as uniform as possible.
- The product's generated stress should be in the compressive stress category.
- Whenever possible, avoid concentration of stress.
- UBE Industries, Ltd. is available for consultation regarding specific design of products.

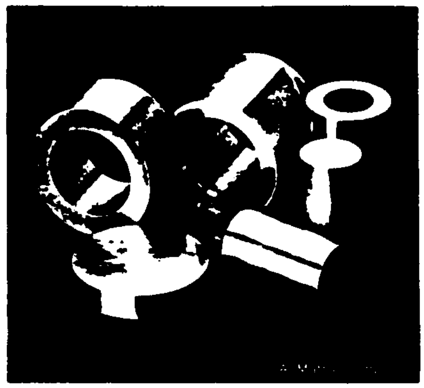
Table 4

Silicon Nitride Whisker

FRP, FRM and FRC have already been developed for various industrial applications, including state-of-the-art materials for space and aircraft uses, materials for automobiles, sporting and leisure goods as well as various other industrial materials. Whiskers are attracting a lot of attention as a material with strong reinforcement capability needed for these applications. And in the technological forefront as usual, Ube has already established a technology for manufacturing silicon nitride whiskers through an imide-decomposition process. Two types of whiskers with different crystal forms are available.

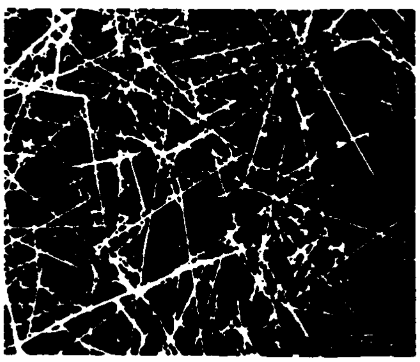
Silicon Nitride Whisker α-Type "UBE-SN-WA"

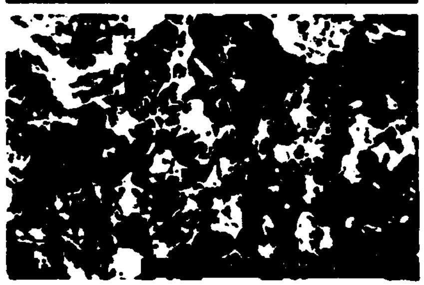
Ube possesses a large-scale Si_3N_4 powder manufacturing plant, which can also produce Si_3N_4 whiskers. This enables economical mass production of high-quality whiskers which in turn makes possible a stable supply according to customer demand.



Quality

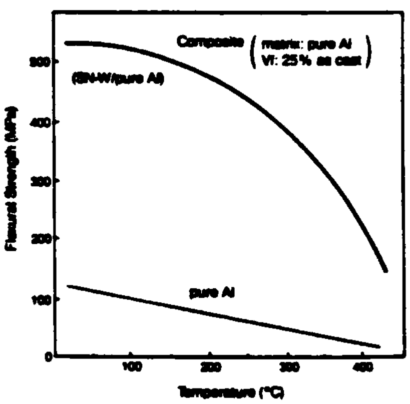
Diameter (μm)	0.1~0.4
Length (μm)	5~20
Aspect ratio (L/D)	20~100
Crystal type	α-type with minimum crystal defects
Purity	Fe 4000~5000 ppm Al <100 ppm Ca <100 ppm O 2~3%





Applications

- Fillers for a variety of composite materials (FRP, FRM, FRC, etc.) including silicon nitride whisker-reinforced aluminum composites
- High-temperature thermal insulation materials
- Whisker-mixed plasma spray coating materials



Flexural Strength of Fiber-Reinforced Metal (Al)

Table 5

Silicon Nitride Whisker β-Type "UBE-SN-WB"

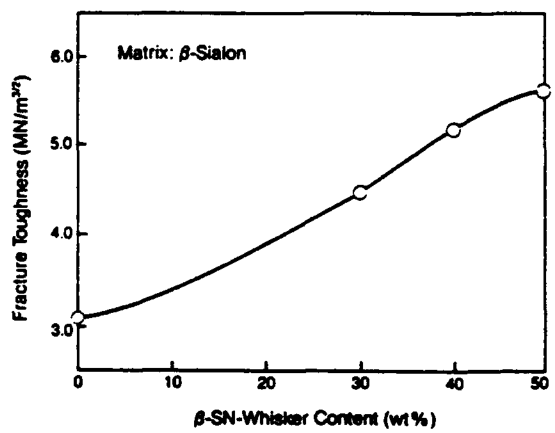
This silicon nitride whisker was developed ahead of worldwide competition, as a reinforcing material for high temperature-use ceramic composites or special metal composites. Known as the "UBE-SN-WB," it offers even higher stability than the β-type in the high temperature range.

Quality

Diameter (μm)	0.1 ~ 0.5
Length (μm)	10 ~ 30
Aspect ratio (L/D)	20 ~ 50
Crystal phase	β-phase

Applications

- Fillers for a variety of composite materials (FRP, FRM, FRC, etc.)
- High-temperature thermal insulation materials
- Whisker-mixed plasma spray coating materials



Fracture Toughness of Fiber-Reinforced Ceramic

Table 6

Tyranno Fiber® UBE

Many fiber-reinforced plastics (FRP), metals (FRM) and ceramics (FRC) are currently being produced. Such fiber-reinforced materials are used as structural materials for industrial products as well as for sports and leisure purposes. Their applications also extend to state-of-the-art purposes including the high-tech materials required for space aircraft. Since the combined fibers used for FRM or FRC come into contact with high-temperature melting metals or ceramics, they must meet especially stringent specifications. Among these requirements are thermal resistance, wetting capability and deterioration resistance.
Ube has developed a new type of continuous inorganic fiber, called the TYRANNO FIBER®. The new fiber is composed of silicon, titanium, carbon and oxygen from an organic metal polymer.

Typical Properties of TYRANNO FIBER

Filament Diameter	8.5±0.5 micron 10.5±0.5 micron
Filaments/Yarn	400, 800, 1600
Density	2.3 — 2.4g/cm^3
Tensile Strength	280kg/mm^2 2.74 GPa 400 KSI
Tensile Modulus	21,000 kg/mm^2 206 GPa 30 MSI
Elongation to break	1.4 ~ 1.5%
Specific Resistance	~10^3 ohm·cm
Thermal Expansion Coefficient (along fiber axis 0 — 500°C)	3.1×10^{-6}/°C
Specific Heat	0.19 cal/deg (60°C) 0.28 cal/deg (400°C)
Heat Resistance (more than 95% strength remains after 6 hours exposure)	1,300°C (in N_2 gas) 1,000°C (in Air)
Resistance to acueous acid and bace NaOH (30%), HNO_3(3N), H_2SO_4(18N), HCl(6N)	any change couldn't be detached after 24hrs dipping at 80°C

Tyranno fibers are available in 400, 800, or 1600 filaments/yarn. The density is 2.37 g/cc, the tensile strength is 2.8-3 GPa (more than 400 KSI), and the tensile modulus is 180-200 GPa (about 30 MSI). Elongation to break is about 1.5 percent. Heat resistance is very good: 95 percent strength retention after 6 hours in N_2 at 1300°C and in air at 1000°C.

The specific resistivity of Tyranno fibers is about 10^3 ohm-cm, but apparently it can be varied from 10^6 to 10 ohm-cm by changing the pyrolysis conditions. In Fig. 2 the temperature dependency of the electric resistivity for Tyranno fibers with different grades is plotted. A through F in Fig. 2 represent the materials with the highest to the lowest resistivity, in descending order.

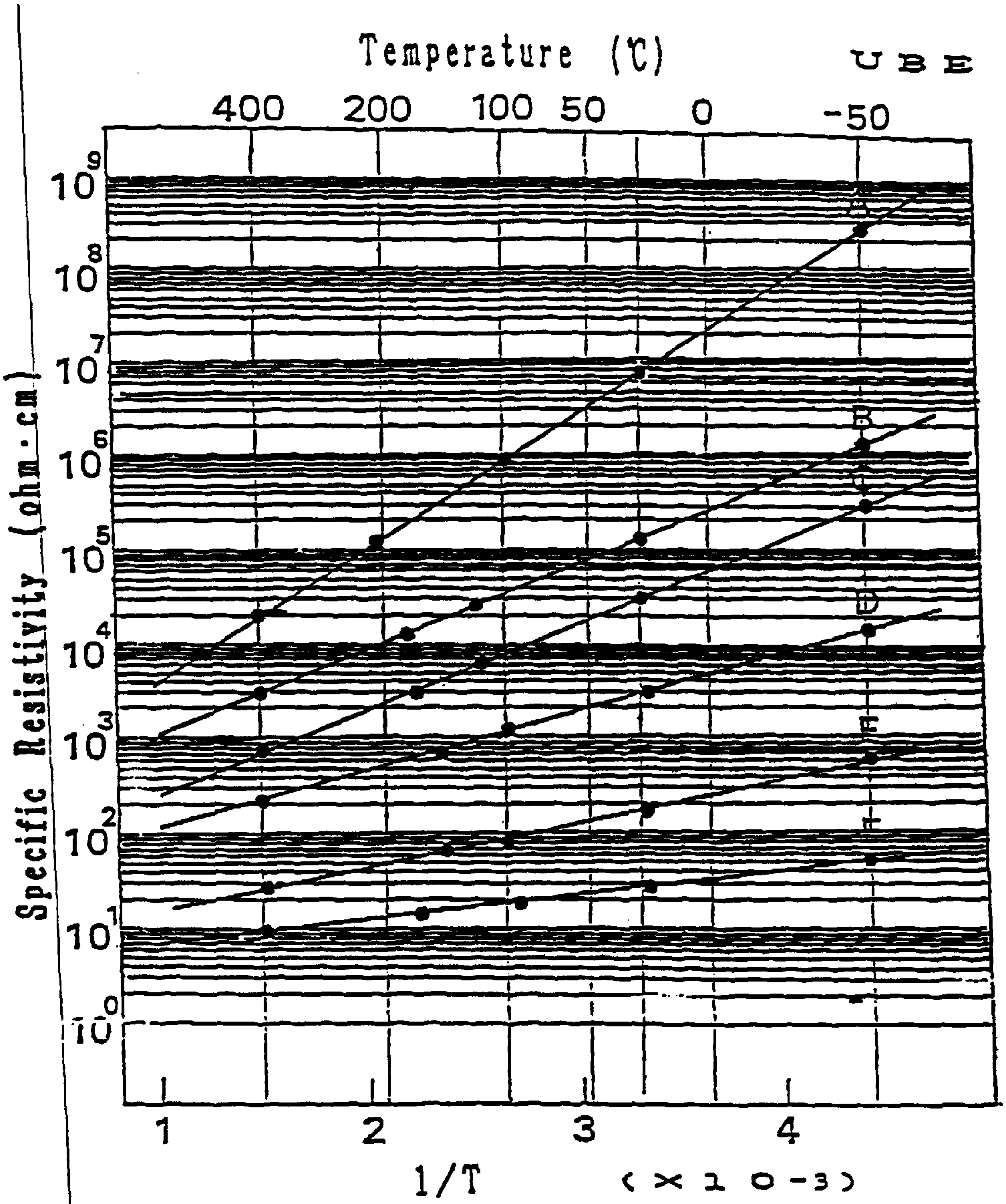
An Ube engineer, Dr. Yamamura, conducted research on inorganic polymer-derived SiC fibers under Professor Yajima of Tohoku University in the early 1980s. The technology was transferred to Ube in 1983, and laboratory production started in 1986. Current production of Tyranno fibers in a semicommercial production plant is about 50 kg/month. Scaleup plans are modest, maybe to 6 tons/year if the market grows. The current market is one-third to Japan (mostly for sporting goods such as golf clubs and tennis rackets) and one-third each to Europe and the United States for aerospace R&D and testing. Textron Specialty Materials is the U.S. supplier of Tyranno fibers.

Tyranno fibers are available in wine red and cobalt blue (Table 7), making them attractive to the sports and leisure markets. They can easily be woven into complex fabrics and have been proposed for suits for firefighters. Tyranno fibers are also available as hybrid fibers, with ceramic powder or whiskers adhering to the surface of the fibers to keep the fibers separated and assure that the matrix can flow between all the fibers, thus reducing voids and assuring high performance.

Ube reports that a unidirectional epoxy matrix composite using Tyranno fibers instead of graphite fibers exhibits 25 to 40 percent increases in compression strength, flexural strength, and transverse tensile strength.

4. Metal Matrix Composites

Ube reported that one cannot obtain rule-of-mixture strengthening of conventional Al alloy matrix/Tyranno fiber-reinforced composite prepared by squeeze-casting because some alloying elements, such as Zn or Mg, attack the fiber. During the past few years MITI has had a national program to identify or develop a new alloy that has the least



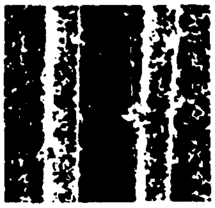
The relationship between specific resistivities of Tyranno fibers and measurement temperature.

Figure 2

Table 7

SPECIALITY GRADES

1. Hybrid fiber



Fine ceramic pulverized powder composites:
Fine ceramic powder is distributed over the surface of the Tyranno fibers in a dot formation.

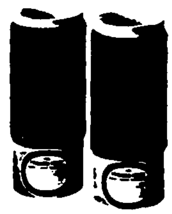
Ceramic Whisker Composites:
Ceramic whiskers adhered to the surface of Tyranno fibers.

Because of its superior performance, Tyranno fiber is expected to be of great use in composites with metals, ceramics or plastics serving as the matrix.

A key step in making successful composites is the even dispersement of fibers and whisker into the matrix. Ube has developed various methods to perfect this process. In cooperation with the Toyota Central Research & Development Inc., Ube has moved to the forefront of the field of fiber-reinforced composite technology through the development of the hybrid fiber.

The hybrid fiber allows fine ceramic particles with diameters of 1/100 to 1/10 of a micron to adhere to the surface of Tyranno fibers. By utilizing this hybrid fiber, the matrix can flow between each fiber, connecting them without any void. In this way a high-performance composite is formed.

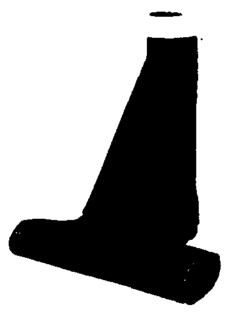
To supplant or just supplement ceramic particles, Ube has also developed fibers with whiskers attached. Utilizing these fibers dramatically improves the performance of composites in a 90° direction to the Tyranno fiber.



In addition to use with Tyranno fibers, hybrid technology is effective when used with graphite or organic fibers and when used with matrices consisting of either metals, ceramics, or plastics. Due to the versatility of this hybrid technology, further developments include the following:

2. Fabrics

The unique characteristics of Tyranno fibers include softness, due to their amorphous structure, as well as a comparatively great elongation (1.5 — 2.0%) and thinness (8.5 microns in diameter). These qualities allow Tyranno fibers to be woven into various types of complex fabrics.



1) Flat fabrics
Fabrics with a wide range of fiber densities from 100g/m² to less than 1000g/m².

2) Tube-shaped fabrics
Tube-shaped elastic fabrics in blade condition.

3) Three-dimensional fabrics
The key fabrics for developing composites for structural materials or heat insulating materials.

3. Colored Fiber

Physical Properties of Colored Tyranno Fiber

Color	Wine Red or Cobalt Blue
Filament Diameter	8 ~ 12 (μm)
Filaments/Yarn	200·n (n = 1 ~ 8)
Density (at 25°C)	2.3 ~ 2.4
Tensile Strength	2.1 ± 0.1 (GPa)
Tensile Modulus	200 ± 10 (GPa)
Tensile Strain to Failure	1.0 (%)
Specific Resistance	8 ~ 13 (× 10^{4}Ω·cm)

Tyranno colored fibers are produced without the use of dyestuffs or pigments, allowing them to retain their color at temperatures up to 1300°C. It is possible to use them alone, with different colored Tyranno fibers or with other fibers. Because of their excellent performance and appearance, this superfine material promises to be extensively applied to products related to sports and leisure, as well as audio and video products.

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deleterious effects at the fiber boundary. It was found that Al-Ni (Ni: 4 to 9 weight percent) and Al-Ti (Ti: 0.5 to 1.5 weight percent) alloys indeed exhibited minimum reaction with the Tyranno fiber surface and maintained near rule-of-mixture strengthening.

Ube's work in metal matrix composites (MMCs) (Tables 8, 9, Figs. 3-8) has been confined to using hybrid Tyranno (Tyranno fibers with ceramic powder or whiskers adhering to the surface of the fibers to keep the fibers separated and assure that the matrix can flow between all of the fibers--a technique developed and used by Toyota) or Si_3N_4 whiskers in combination with matrices of Al-Ni, 6061, and other Al alloys. Processes include squeeze-casting for small, complex shapes and preparation of sheet prepreg by plasma spraying preforms for large shapes that are hot-pressed (diffusion bonding).

Apparently either SiC powder or Si_3N_4 whiskers can be used to prepare hybrid Tyranno, and at 55 fiber volume percent the longitudinal and transverse strengths are twice those realized with untreated Tyranno fiber.

Hybrid Tyranno/Al suffers no loss in flexural strength after 400 hours at 623°K, and bending fatigue strength after 10^7 cycles is about triple that of 6061-T6.

Ube has studied Al-Ni alloys with 2, 4, 6, and 8 percent Ni; flexural strength increased from 30 to 45 kg/mm^2 at 8 percent Ni.

5. Ceramic Matrix Composites

Ube reported on its Tyrannohex ceramic matrix composite (CMC) for the first time at the 1989 Cocoa Beach meeting of the American Ceramic Society. Tyrannohex CMC is still under development and evaluation, and hence no literature is available.

The history of Tyrannohex is interesting and descriptive of how adequate staffing, funding, and persistence can contribute to successful innovation. With Tyranno fibers in hand, Ube decided to make CMCs. They tried every conceivable candidate matrix with little success, so they focused only on the most promising matrix, varying the fiber content over very wide ranges. Less and less matrix gave better composites, so Ube eventually settled for no matrix. Tyrannohex is made by laying up webs of Tyranno fibers in unidirectional or crossply layups and hot-pressing at about 2000°C and 700 atmospheres. Ube says that the oxygen begins to volatilize as CO at about 1500°C and is gone by 1800°C. The round Tyranno fibers are converted to hexagonal cross section, and the resultant "ceramic wood" breaks at a strain of about 3 percent. Ube claims no strength loss at room temperature after 1 hour of exposure to 1700°C in air.

Table 8

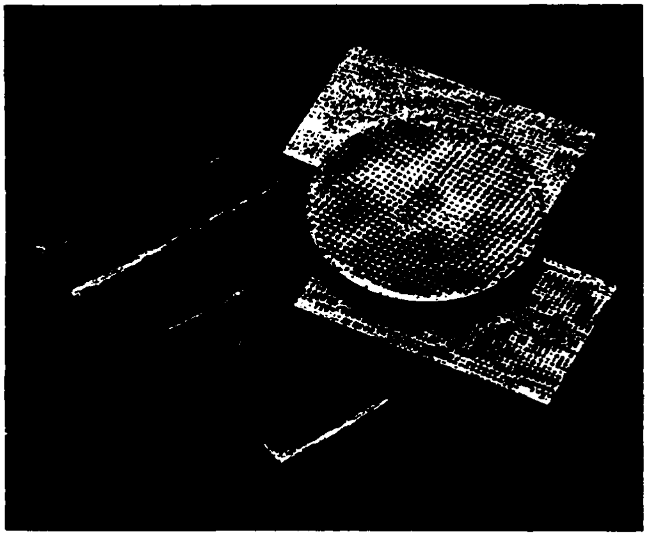
Metal Matrix Composites UBE

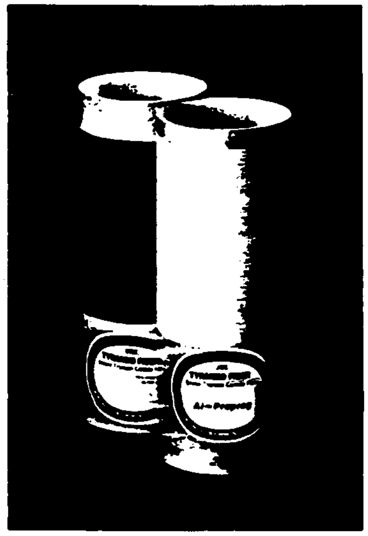
Fiber-Reinforced Metal Composites (FRM) are now being developed in many different technological fields. Now a high-quality FRM, a cut above conventional FRMs, has been developed by Ube. Ube's superior FRM was produced by using original technology to develop reinforcing materials such as "Tyranno fiber" and "silicon nitride whisker."

In addition, Ube has also produced metal composite materials (such as magnesium) as well as a die casting machines and even molded products.

Currently, Ube's FRM activities have focused on the production of reinforcing materials such as "Tyranno fibers" and "Silicon Nitride Whisker." By combining these materials with carbon fibers or ceramic materials the development of hybrid reinforcing materials is progressing.

Molding methods employed by Ube include die casting, squeeze casting as well as hot pressing methods.





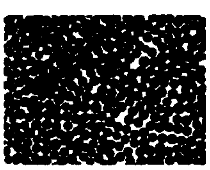


Photo 1: Cross section of FRM molded product.
Each Tyranno fiber (represented by the black circles) is thoroughly dispersed throughout the metal No clusters of fibers may be present

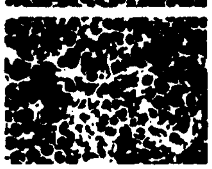


Photo 2: Fracture surface of Tyranno FRM (fiber section)

Photo 3: Fracture surface of Tyranno FRM (section parallel to the fibers)

Properties

When FRM are molded using Ube's original hybrid Tyranno fibers, the fibers are easily dispersed and the metal fills the spaces between the fibers, leaving no void.

The performance of the FRM molded product is directly related to the bonding between the fibers and metal. When the fiber and metal types are incompatible, the wetting property between them is impaired, resulting in frequent occurrence of voids.

If the wetting property is good, but no adhesive force exists between the fiber and metal, the fibers will fall out and be deformed by external force, reducing the strength of the molded product.

On the other hand, if the metal and fiber are extremely compatible, the dissolved metal and fibers will be reactants to each other at the molding stage. This makes the adhesion too strong which in turn will decrease the performance capability of the fibers and the finished product. Too strong an adhesion will cause the molded products to become more fragile and easily break.

In view of these effects it may be concluded that the ideal condition between the fibers and metals of FRM is one in which they are not reactants to each other yet adhere to an appropriate degree.

By examining the fracture surface of Tyranno FRM (Photo 2), you can see that the fibers have not completely fallen out and that the matrix metal is slightly transformed. (Fracture not a result of fragility.)

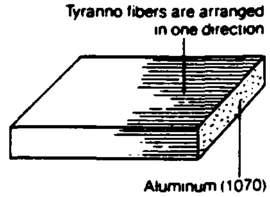
Photo 3 shows the fracture surface in a direction parallel to the fiber. You can see by the marks that the matrix aluminum adheres to the fibers. (If aluminum does not adhere to the fibers, a smooth fiber surface will be observed.)

Table 9

Mechanical Properties of FRM

1. Value at Normal Temperature

	FRM (Tyranno fiber plus aluminum)	Aluminum (1070)
Flexural strength in the direction of fiber axis	150kg/mm^2	—
Tensile strength in the direction of fiber axis	110kg/mm^2	10kg/mm^2
Flexural strength in the direction 90° to fiber axis	40kg/mm^2	—



Vf = 50% (Amount of Aluminum/Amount of Tyranno Fibers)

2. Properties of FRM and reactivity with Al alloy

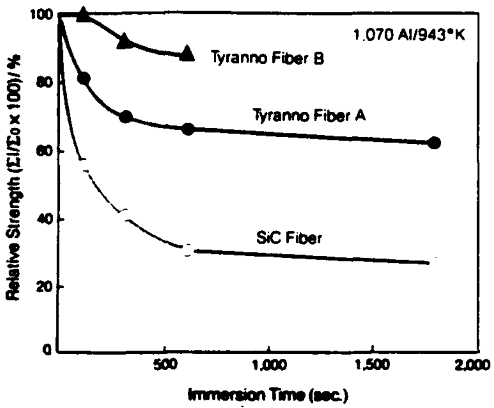


Fig. 2-1 Reactivity with Melting Aluminum (Tyranno Fiber B Contains more Ti than Fiber A)

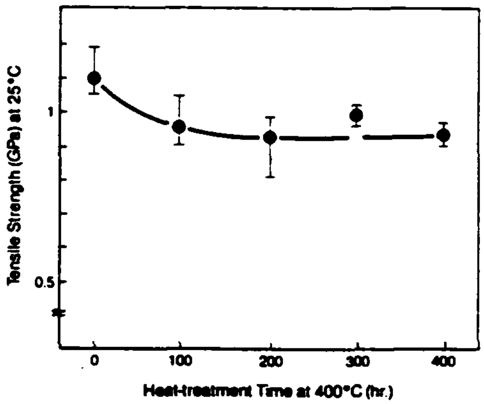


Fig. 2-2 Thermal Stability

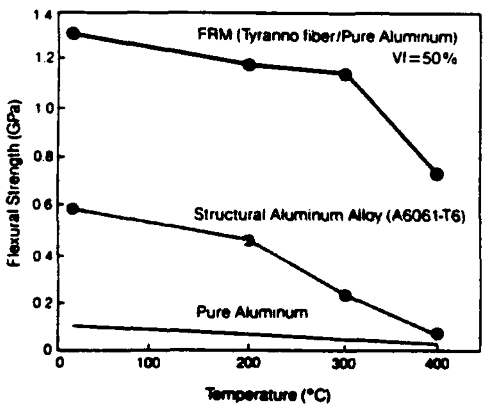


Fig. 2-3 Temperature Dependence of Flexural Strength

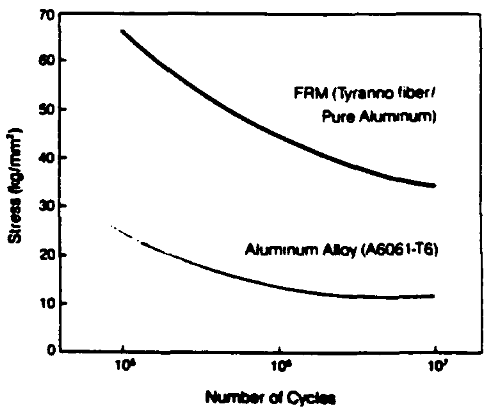


Fig. 2-4 Fatigue Strength

UBE INDUSTRIES, LTD. Corporate Research & Development Division

Market Development Department: ARK Mori Building, 12-32, Akasaka 1-chome, Minato-ku, Tokyo, 107 Japan
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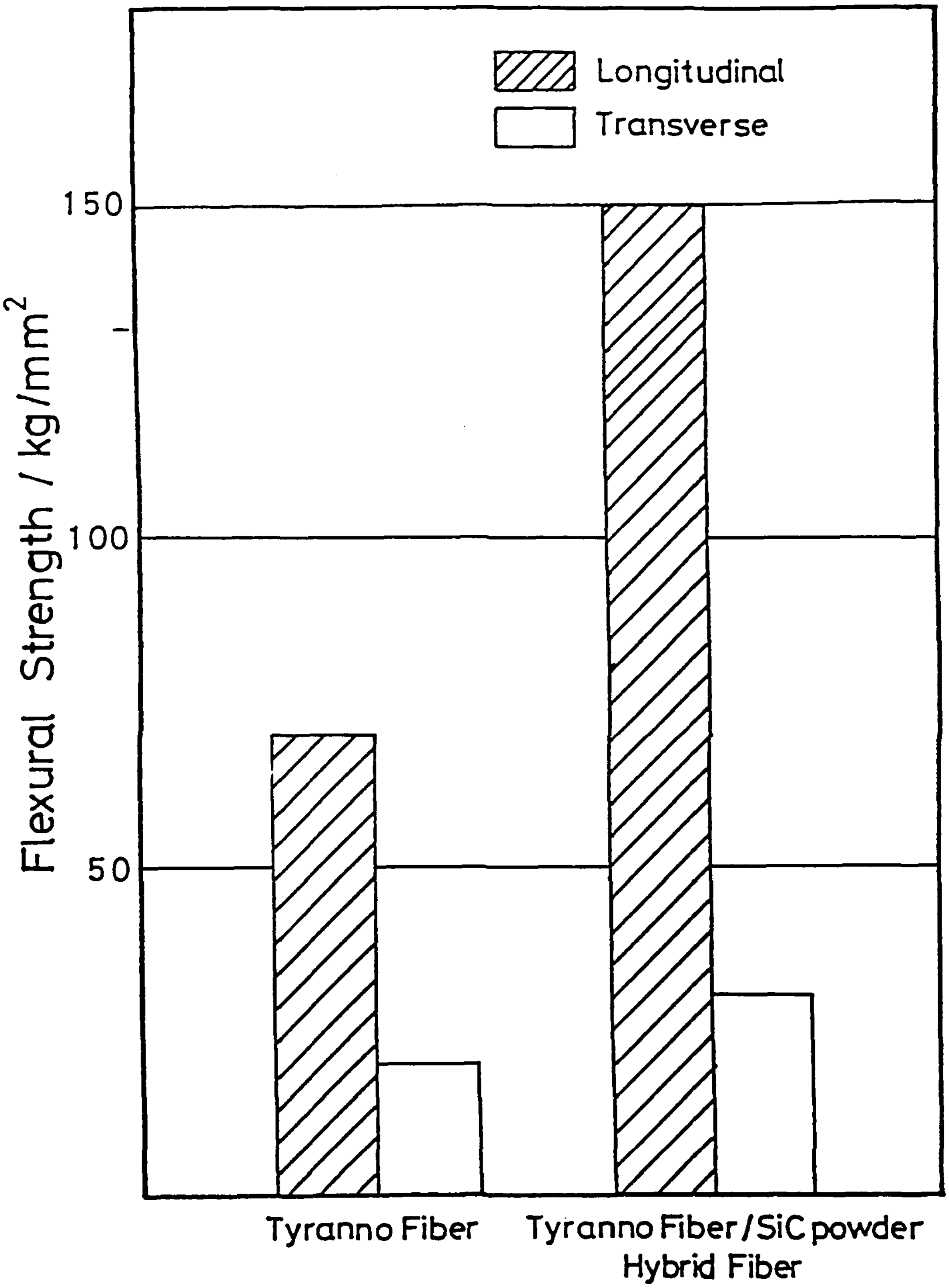

Longitudinal
Transverse
150
100
50
Flexural Strength / kg/mm^2
Tyranno Fiber
Tyranno Fiber/SiC powder
Hybrid Fiber

Figure 3

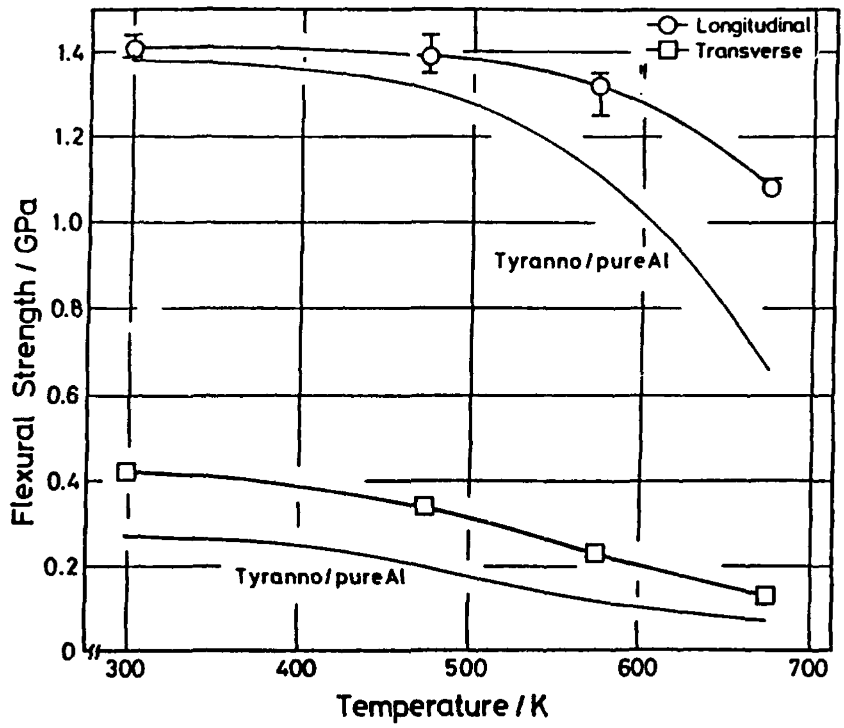


Figure 4

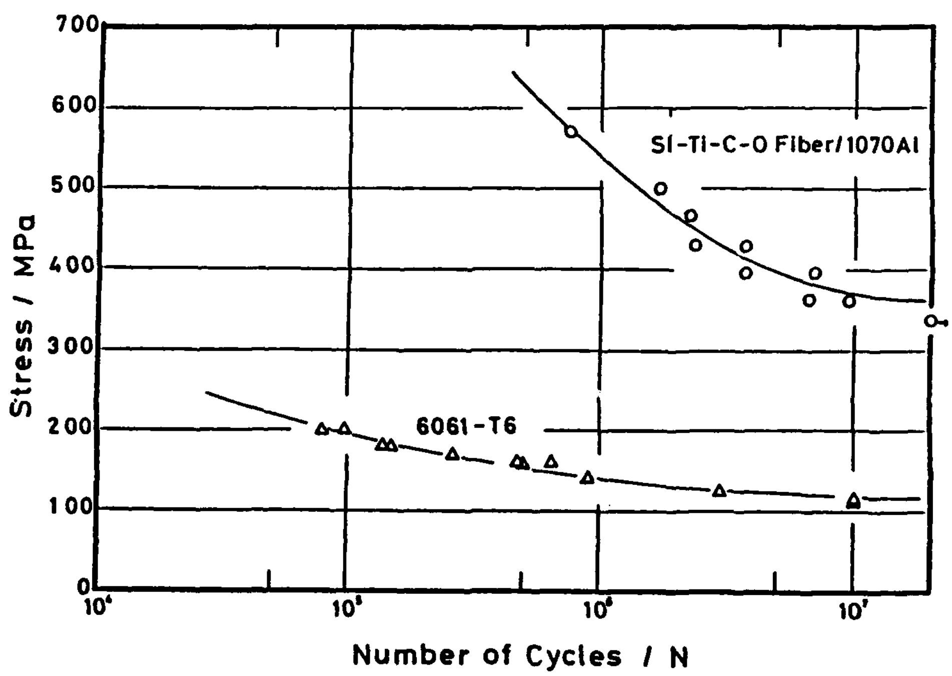
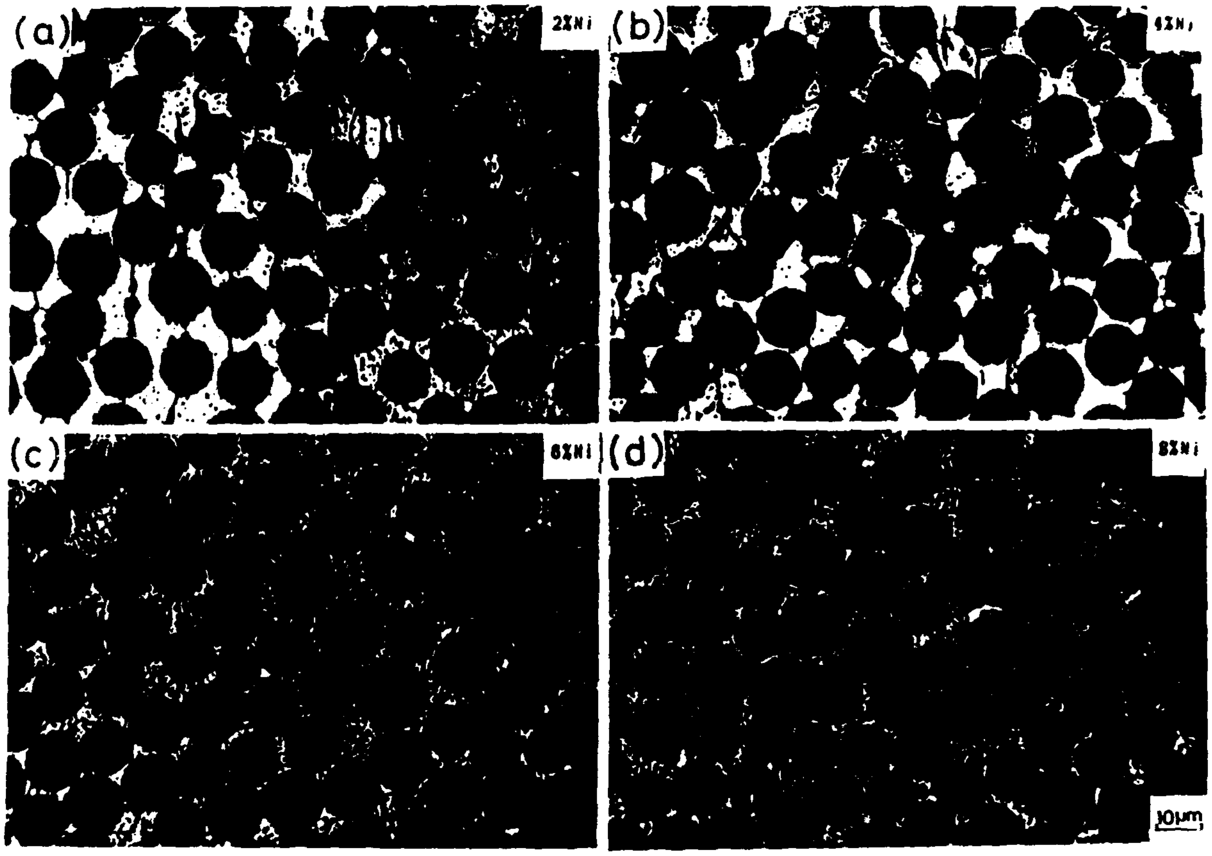


Figure 5



Microstructures of Transverse Cross Sections of Tyranno Hybrid Fiber-Reinforced Al-Ni Al-Ni Alloy

Figure 6

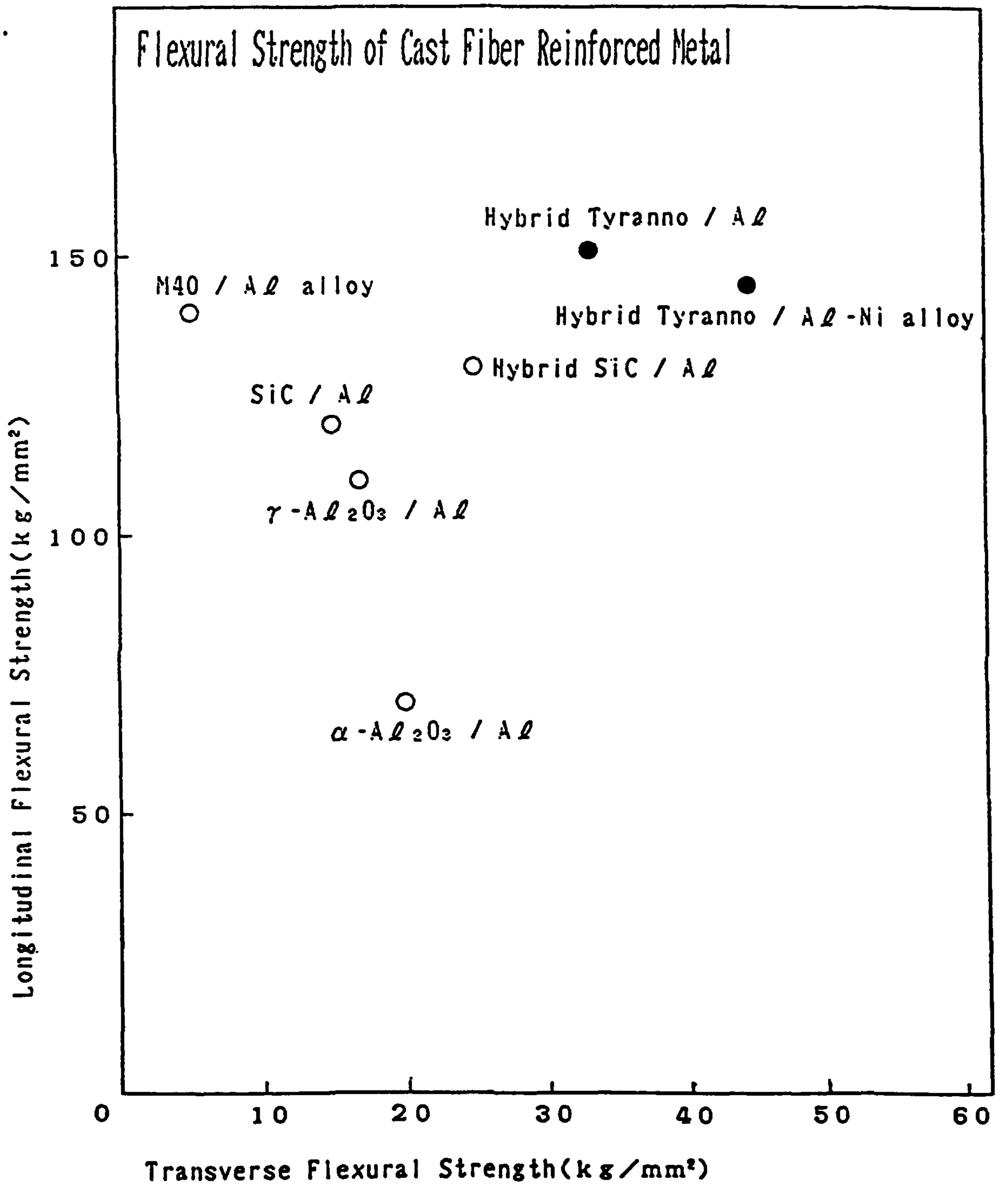
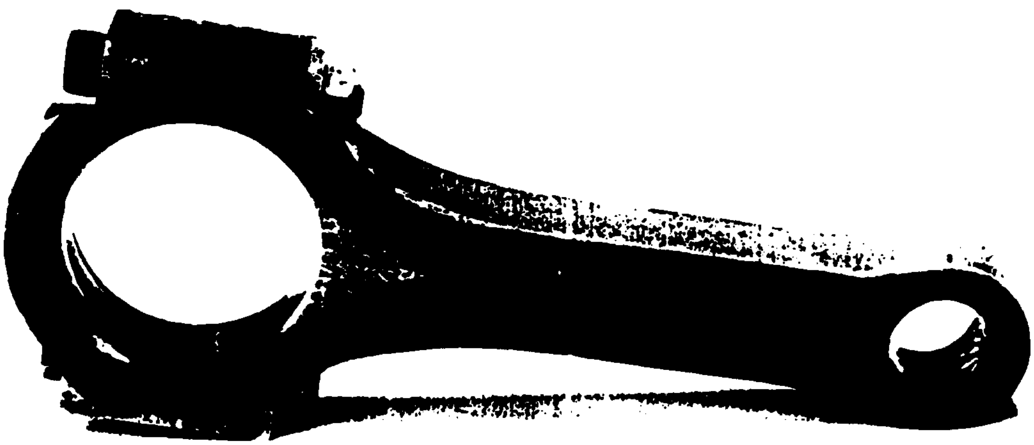

Flexural Strength of Cast Fiber Reinforced Metal
Hybrid Tyranno / Aℓ
M40 / Aℓ alloy
Hybrid Tyranno / Aℓ-Ni alloy
Hybrid SiC / Aℓ
SiC / Aℓ
γ-Aℓ₂O₃ / Aℓ
α-Aℓ₂O₃ / Aℓ
150
100
50
0
10
20
30
40
50
60
Longitudinal Flexural Strength(kg/mm²)
Transverse Flexural Strength(kg/mm²)

Figure 7



Hybrid FRM Connecting-Rod

Figure 8

6. Tours, Discussions, and Conclusion

In addition to the video tapes, oral presentations, and literature handouts described above, there was an auto tour of the wide array of Ube Industries facilities in the Ube City area, and there were walking tours of the silicon nitride powder plant, the Tyranno fiber plant, and the Ube Scientific Analysis Laboratory. Two impressions were prominent: (1) cleanliness and order, and (2) company pride and loyalty. As the days and visits progressed, both impressions were reinforced elsewhere, but they seemed more intense at Ube Industries, maybe because it was the first exposure to Japanese industry for most of the TAT members.

It is difficult to express the impression of cleanliness and order in toto, but it was manifested in the following ways, among others:

- Every concrete plant floor was not only clean but brightly painted, with safe walkways clearly designated.
- Employees and management were virtually indistinguishable because all wore a common uniform (neat, quite well fitting, and clean) with no evidence of role or rank distinguishable.
- Employees and visitors had to leave their shoes at the door and wear special slippers indoors (even in the offices at many companies).
- Processing facilities (for handling and mixing powders, for drawing fibers, etc.) were usually in clean rooms, and entry into the clean rooms to observe the processes often required donning disposable pants, jackets, and caps.

During the concluding discussions staff members observed that Ube expected to develop MMC and CMC processes and products by itself but was ready to license technology and to sell fibers to all comers. At present the Japanese market is a commercial market with little application other than high-technology sporting goods (tennis rackets, golf clubs, racing car bodies, special sailboats, etc.). The people at Ube expect it may be the next century before large markets for MMCs and CMCs appear. (All over Japan the emphasis on commercial markets and long-range planning stood out.) One recent development may dramatically change the current emphasis, however. In January 1989 the Japanese Diet approved a new MITI program for $80M over 8 years. (Since Japanese government R&D funding usually covers only materials and some operational costs, but not manpower or major equipment costs, the industry contribution may be 10 times greater.) The just-concluded MITI program had a composites thrust--for commercial

applications. The new MITI program emphasizes not only high performance but also high-temperature capability (1700°C in air for short times), and at Ube and most other companies the TAT visited there was great enthusiasm for a role in the program. The most notable objective was to provide materials technology for the spaceplane HOPE, which is to begin development in 1992-1993, but a general advance in aerospace technology seems to be in the air. This thrust will include Ti and intermetallic matrix composites and C-C structural materials as well as new high-temperature fibers.

In summary, Ube gives an impression of aggressive vitality, and it has several quality products of interest to the U.S. defense community, among them the Si_3N_4 powder, Tyranno fibers, Tyrannohex, and new MMC technology.

B. KOBE STEEL, LTD. (KOBELCO)

The TAT visited Kobe Steel, Ltd. (KOBELCO) on 1 February 1989 in Kobe City, Hyogo Prefecture. KOBELCO is one of the world's top 20 producers of iron and steel products and one of the most diversified corporations in Japan. Its products and services include industrial and construction machinery, new materials, plant engineering, and finished and unfinished ferrous and nonferrous metal products. Iron and steel sales in 1987 totaled ¥453 billion, machinery ¥269 billion, aluminum and copper ¥213 billion, and welding ¥41 billion, for a total of about $7.8 billion.

KOBELCO is in the midst of large changes. As part of its new globalization strategy it opened three local production projects in 1987 in the United States--Kobe Copper Products, Inc. in Greensboro, North Carolina; Kobe Precision, Inc. in Silicon Valley, California; and Kobe Metal Powder of America in Indiana. They join 12 other KOBELCO overseas manufacturing plants and many overseas subsidiaries and trading companies. There are approximately 80 KOBELCO plants in Japan.

Kobe research facilities are in West Kobe and Tsukuba Science City in Japan, in London, and in Research Triangle, North Carolina. In 1987, 3.3 percent of net sales was spent on R&D. The principal research areas were electronics, biotechnology, extreme physical properties technologies [superconducting wire and magnetic systems, high-temperature (HT) superconductors, hot and cold isostatic presses (HIP and CIP), etc.], surface treatment and superprecision machining, and new materials (shape memory alloys, powder metallurgy and advanced composites, including MMC and C-C). The introductory video shown the TAT mentioned new materials and new processes again and again. MMC was noted as a newcomer to the aerospace and automotive industries. Surface finishing technology has emphasized resistance to corrosion, heat, and wear; several highly polished surfaces for copiers and printers were shown. There were many pictures of clean rooms and covered personnel. Thin film technology using an excimer laser and diamond film technology were highlighted. Superconductivity research began in the 1960s and is now getting emphasis. As evidence of KOBELCO diversity, the video showed a coal liquefaction pilot plant in Australia, sporting goods incorporating C fibers made from pitch by KOBELCO, and a computer-controlled excavator to illustrate KOBELCO's machinery and software capabilities.

1. Carbon-Carbon

Research in carbon/graphite materials began about 10 years ago and has been focused on carbon-carbon (C–C) composites R&D in recent years. The KOBELCO target application is aircraft brakes. No figures on production or costs were available, since there has been no production so far. Trial developments suggest 600 kg/year production is possible, producing about 800 plates/year.

KOBELCO was not very explicit about its C-C process. For example, it was unclear whether KOBELCO uses T300 PAN fibers or its own pitch fibers for the heavy felt it starts with. At one point it seemed that spokesmen said that pitch was added to the fiber preform by chemical vapor deposition (CVD), but all later discussion and conversation suggested initial impregnation with resin into an evacuated preform, precarbonization, resin impregnation, carbonization, then subsequent HIP impregnations with pitch and a final high-temperature carbonization. Five impregnations, the last three at 500 to 1000 atmospheres (to get higher density than with vacuum), give 1.4 to 1.7 g/cc density. Nondestructive evaluation (NDE) via ultrasonics and soft x ray is augmented by mechanical property tests to assure quality.

Overall, KOBELCO reports the mechanical properties of its C-C to be isotropic, and it claims short production times, quicker delivery, and lower cost because the KOBELCO process requires fewer impregnations. KOBELCO also claims more varied properties are possible because it can impregnate at the high-pressure stages with glass or metals such as Cu or Al. KOBELCO claims to control the electrical and thermal conductivity in Cu/C-C and also reports the use of Cu/C-C in bushings and electric motor brushes.

The attached data sheet (Table 10) provides information on the KOBELCO C-C materials. CFC10H is C-C with nominally 10 percent residual porosity. CCM30Cu is C-C with 30 percent porosity filled with Cu.

2. C Fibers

Apparently KOBELCO has studied production of both PAN-based and pitch-based C fibers but now is focusing on coal tar pitch-based fibers. KOBELCO is running a small-scale (100 kg/month) plant to develop production technology and is doing a feasibility study for a 50-100 tons/year pilot plant. KOBELCO is spinning C fibers from mesophase pitch; they oxidize in air at 300°C, carbonize in N_2 at 1000°C, and graphitize in He or Ar at

Table 10

1. General Characteristics

	CFC10H	CCM30Cu	CCM30Al	Method
Bulk density, g/cm^3	1.60	3.67	1.80	JIS R7212
Specific weight, g/cm^3	1.90	4.00	1.87	JIS R7212
Porosity, %	16	8.3	4	Computed value
Open pore volume, cc/g	0.20	0.03	0.07	Mercury porosimeter

2. Mechanical Strength

		CFC10H	CCM30Cu	CCM30Al	Specimen size, mm
Flexural strength, MPa	⊥	200	150	180	10×50×3 (t) 3-point bending
	//	150	140	—	
Flexural elasticity ratio, GPa		30	20	40	10×50×3 (t) 3-point bending
Compressive strength, MPA	⊥	200	300	400	6×6×10 (t)
	//	130	200	200	
Shore hardness	⊥	65	60	80	
	//	45	60	—	
Izod impact value, J		0.5	0.5	—	10×50×10 (t) (2.54V notch)

3. Electrical and Thermal Properties

		CFC10H	CCM30Cu	CCM30Al	Method
Electrical resistance, mΩcm		3.0	0.15	0.2	Four-terminal method
Specific heat, cal/g°C	R.T.	0.19	0.13	0.22	Laser flash method
	1000°C	0.50	—	—	
Thermal conductivity, kcal/mHr°C	R.T.	6.5	18.6	5	Laser flash method
	1000°C	8.7	—	—	
Thermal expansion coefficient, $\times 10^{-6}$/°C R.T.–700°C	⊥	8.0	7	12	Helium atmosphere
	//	0.3	1	2	

4. Producible Sizes, mm, max.

	CFC10H	CCM30Cu	CCM30Al
Disk, dia.×t	280×30	90×30	90×30
Board	200×200×30 t	80×320×30 t	80×300×30 t
Applications	Brakes	Pantographs Motor brushes	Seals for pumps and compressors

>2500°C. KOBELCO can vary the crystallite orientation from random to highly radial but seems to prefer the random. For 12 μ fibers in 1000 filament tows produced at 10,000 tons/year, KOBELCO has target prices of \$20/lb for middle-performance fibers (200 kg/mm^2 and 20 tons/mm^2), \$40/lb for high-performance fibers (400 kg/mm^2 and 40 tons/mm^2), and \$200/lb for ultrahigh-performance fibers (500 kg/mm^2 and 50 tons/mm^2).

3. SiC Whiskers

KOBELCO uses a solid-state reaction (vapor-liquid-solid over a Ni catalyst) to produce β SiC whiskers with low density, high strength, high modulus, high resistance to heat and water, and high chemical stability. KOBELCO is currently producing 100 kg/month and is considering a 6-12 tons/year pilot plant. Three types and two grades are presently available. The process is continuous and costs should be low (<\$40/lb). KOBELCO is distributing samples for evaluation, and another part of Kobe Steel is using the whiskers in MMC and CMC R&D.

4. Tour

After a brief plant tour where HIP units are manufactured for sale or use in house (most of the 140 units, ranging from 4 inches in diameter by 10 inches long to 1 ft in diameter by 6 ft long, have been sold, but two are retained for C-C and M/C-C research), the TAT was taken to the new Seishin Research Area for presentations about and laboratory tours of the Applied Mechanics and Electronics Technology Centers. Research in the Applied Mechanics Center focuses on structural engineering, dynamics and acoustics (including noise suppression), and fluid and thermal engineering (including heat transfer and combustion). There is a very strong dependence on computer simulations to replace costly experiments. CASTEM is a casting analysis system that uses analytical modeling to replace the trials and errors of casting research. HIPNAS is a HIP Numerical Analysis System to relate final shape after shrinkage. CIPNAS is a CIP Numerical Analysis System. All of this software is for sale.

The Electronics Technology Center has sections for research on systems control, mechatronics, instrumentation, and electronic applications. Among the projects are a Bucket Control System to control a backhoe with one lever, an Offline Robot Programming System to teach robots by computer, an Expert Forecasting System to decrease heat in blast furnaces, a 3D Analyzing System to generate 3D (stereo) scanning electron microscope

(SEM) images, a Nanometer Characterization System, a Precision Microwave Level Meter to measure the level of molten metal in a hot pot, and R&D on diamond films.

KOBELCO has studied diamond synthesis for 3.5 years and has four people active on the project. They use plasma synthesis and get polycrystalline diamond films with well-defined facets on Si substrates. They can produce single-crystal films for P-N junctions and can get both p- and n-type semiconductors. Their main goals are high-rate deposition and uniformity over large areas. They are conducting R&D with North Carolina State University.

Target applications for the diamond research include:

- Abrasion-resistant coatings on cutting tools, windows, eyeglasses, and speaker diaphragms
- Heat sinks
- Sensors in severe environments (such as in space nuclear reactors)
- X-ray and UV sensors
- Thermistors for auto, aircraft, and rocket engines
- Electronic devices in severe environments.

KOBELCO has not considered diamond films in lubrication systems.

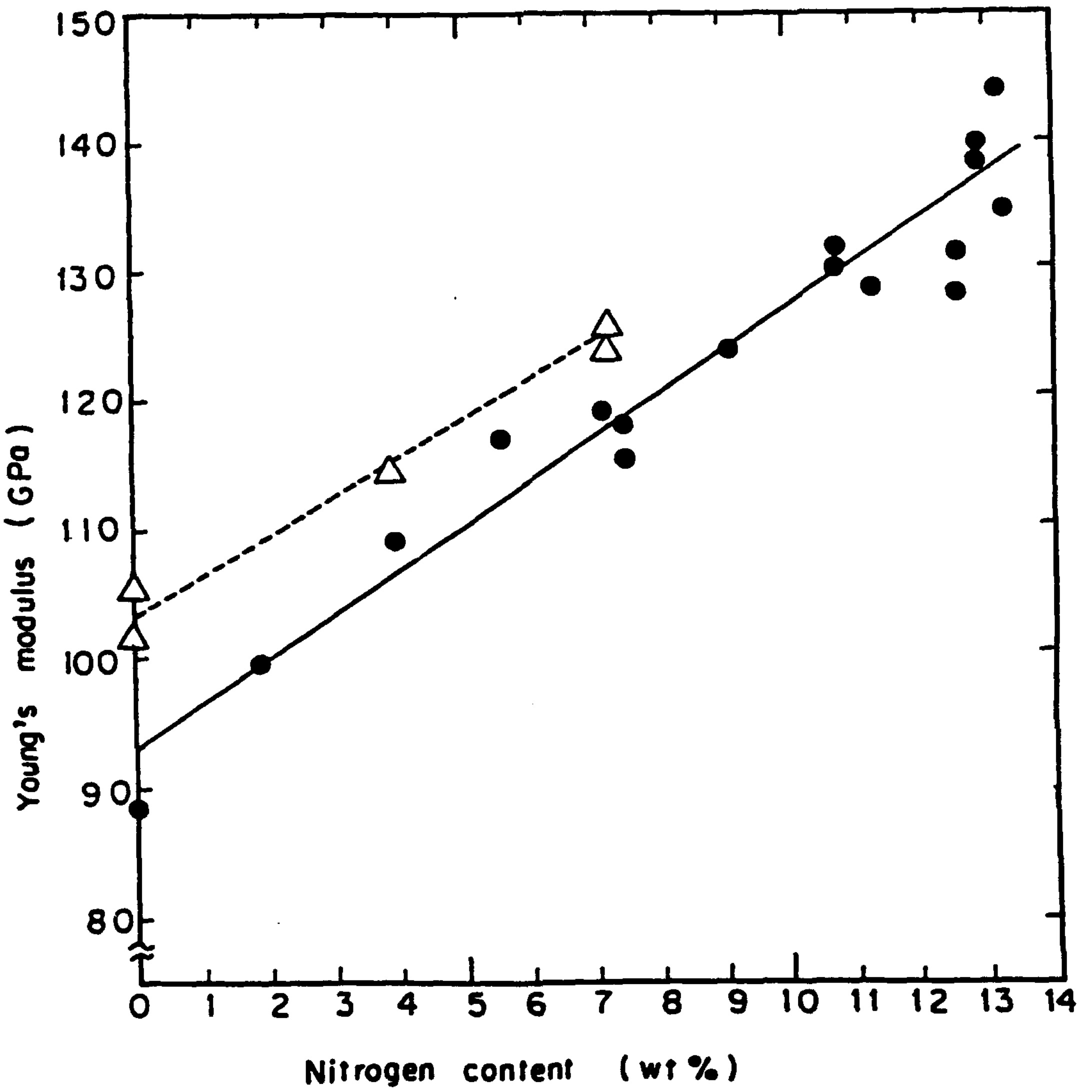
C. SHIMADZU CORPORATION

The TAT visited the Sanjyo Works of Shimadzu Corporation in Kyoto on February 2, 1989. Founded in 1875, Shimadzu has 4000 employees manufacturing analytical and measuring instruments, materials testing machines, process control equipment and instrumentation, medical systems and equipment, industrial instruments and equipment, and aircraft instruments. Shimadzu is number three in the world in medical instruments and in materials testing equipment, and is well known in the United States for heads-up display units, magnetic anomaly detectors, and various mechanical, hydraulic and electrical equipment for aircraft.

Shimadzu has 120 engineers in Kyoto and Tokyo laboratories working on new materials, computer hardware and software, and biotechnology, all of which are considered important to Shimadzu's future. Shimadzu has three overseas subsidiaries, including Shimadzu Scientific Instruments, Inc. in Columbia, Maryland, which manufactures gas chromatographs and other analytical instruments, and Shimadzu Precision Instruments, Inc. in Torrance, California, which markets aircraft and medical equipment.

Shimadzu is not a materials supplier and, though grateful for our interest in their oxynitride glass fiber, its staff members professed surprise and curiosity about the TAT visit, especially since Drs. Lenoe and Pettit had visited them earlier. (The TAT visit and the earlier visit by Lenoe and Pettit were both prompted by extremely intriguing claims about properties and production capabilities in the Japanese trade literature.) They explained their involvement with the fibers as naturally ensuing from their work in ceramic superconductor materials and their development and production of materials testing equipment.

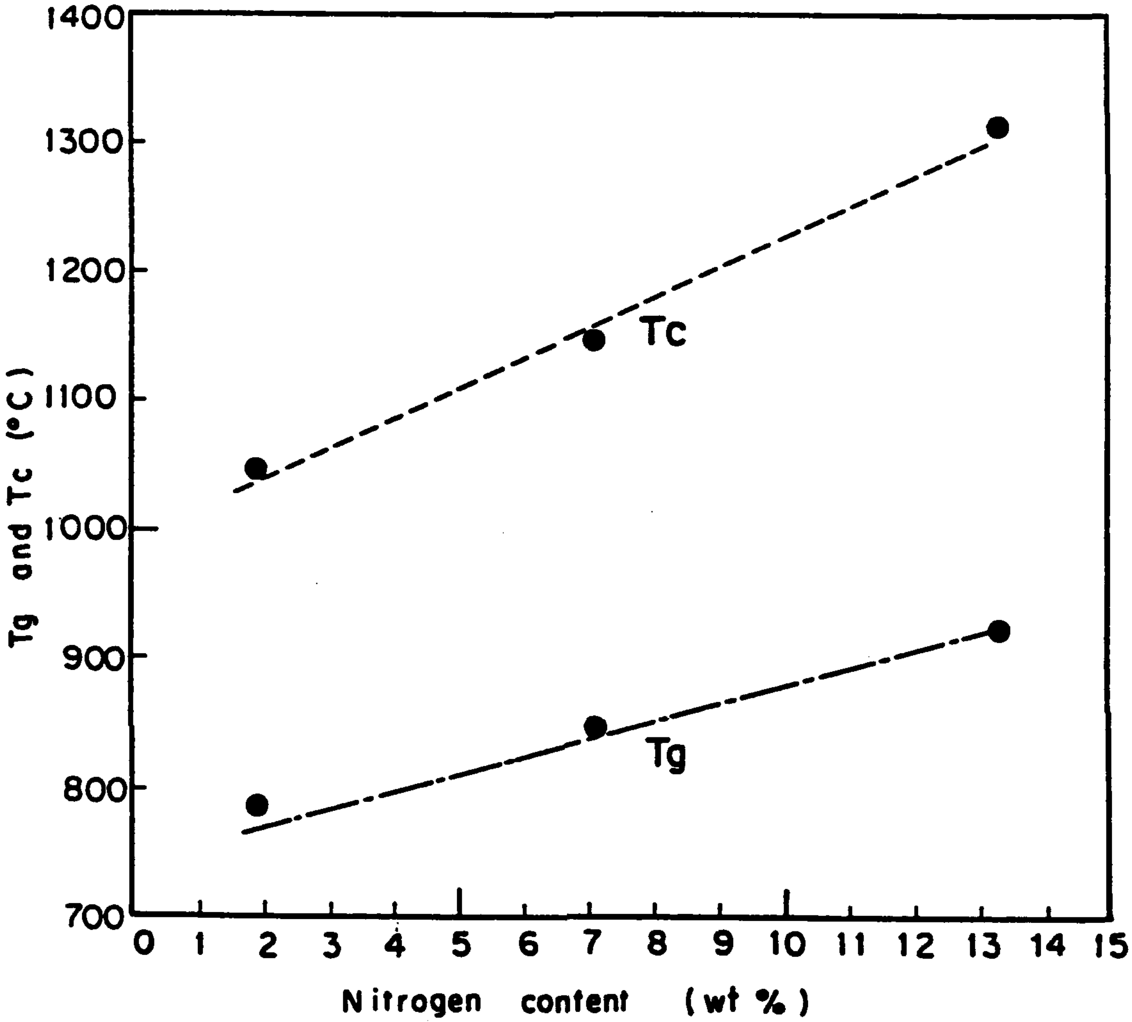
Shimadzu is not presently producing the oxynitride glass fiber. In fact, it is not even in advanced development; Shimadzu expects to spend about 2 or more years on research on the Si-Ca-Al-O-N system. Increasing the N content above 7-8 percent is difficult, but Shimadzu has found how to do it and thereby greatly increase Young's modulus and the glass transition and crystallization temperatures as shown in the attached charts (Figs. 9, 10, Table 11). Most of the fibers have been monofilaments drawn at 500 to 1000 meters per minute. At present Shimadzu is drawing 20 filaments simultaneously but is searching for better bushing materials. In general, Shimadzu seemed to be on a steep slope on the learning curve about glass chemistry, glass processing, and fiber drawing and handling.



Young's modulus as a function of nitrogen content.

Shimadzu Corporation

Figure 9



Glass transition and crystallized temperature as a function of nitrogen content.

Shimadzu Corporation

Figure 10

Table 11

Properties of Oxynitride glass fiber

Sample	Diameter (μm)	Tensile Strength (kg/mm^2)	Tensile Elastic Modulus (kg/mm^2)
Monofilament	5	400~500	18,000~20,000
	15	170~250	13,000~18,000
Strand (200 filaments)	15	200	14,000
Carbon fiber (T300)	7	353	23,000

Shimadzu Corporation

Shimadzu has done a small amount of work in using its fibers in polymeric and metallic matrices and exhibited great interest in the TAT perceptions of eventual markets, maybe somewhere between E and S glass on one side and graphite fibers on the other.

D. TORAY INDUSTRIES, INC.

On February 3, 1989, the TAT visited the Shiga Plant of Toray Industries, Inc., located in Ostu, near Kyoto. Formerly Toyo Koyon, Toray is one of the world's largest and most diversified manufacturers of synthetic fibers and other polymer products. Of almost $6 billion of sales in 1988, 52 percent was synthetic fibers and textiles, 24 percent was plastics, 12 percent was construction and engineering (especially textile machines, data processing systems, and various instruments and equipment), and 4 percent was chemicals. The remaining 8 percent of sales came from the new-product area, which includes pharmaceuticals, photographic products, carbon fibers, composites, and fine ceramics. At present, Toray is engaged in a long-term diversification program that stresses development in electronics, health care products, advanced composite materials, and biotechnology.

1. Carbon Fibers

On the TAT's arrival at the Shiga Plant, the first activity was a tour of the Toray Showroom, where a truly impressive array of Toray products was exhibited. This was followed by a comprehensive exposition of Toray's world view of the PAN-based carbon fiber industry, ranging over a chronology of carbon fiber development, world production capacity, international relationships, world consumption, advanced composites manufacturers in Japan identified by product area, and Toray data on all of its fibers, the manufacturing process, and some composite properties. Many of the charts are reproduced here (Tables 12-20, Figs. 11-15). The TAT did not see the carbon fiber production plant; it is located at Matsumae-machi in Ehime Prefecture on Shikoku Island. R&D on carbon fibers and the manufacture of prepreg are done at the Shiga Plant.

Among the comments noted during the carbon fiber presentation are the following:

- Toray C fibers are very stable because of the low impurity (K, Na) content.
- Fiber surface treatments vary with the strength and modulus of the fiber.
- Toray feels it is reaching the limit in C fiber properties; it is now emphasizing R&D on resins and composite processing.
- The interface limits the benefits of T1000 fibers in polymeric matrices.
- Little effort has been applied to the use of high-performance thermoplastics such as PEEK so far. This will come, but Toray expects processing to be a problem.

Table 12

Chronology of Carbon Fiber Development

Year	Low Grade	High Grade		
		Rayon	PAN	Pitch
1958	UCC (Rayon)	--	--	--
1960	--	--	Shindo Patent	--
1962	Nippon Carbon (PAN)	UCC Patent	--	--
1963	Otani Patent (Pitch)	--	--	--
1964	--	UCC	RAE	--
1967	--	--	Courtaulds Morganite Rolls Royce	--
1968	--	--	--	Otani Patent
1969	--	--	Nippon Carbon	--
1970	Kureha Chemical (Pitch)	--	--	--
1971	--	--	Toray	--
1972	--	--	Hercules	--
1973	--	--	Toho Rayon	--
1974	--	--	--	UCC
---	---	---	---	---

Table 13

World Production Capacity

PAN based		1000 lbs/year
Japan	Toray	3300
	Toho Rayon	3120
	Asahi Kasei Carbon Fiber	660
	Mitsubishi Rayon	260
Taiwan	Taiwan Plastics	220
Korea	Korean Steel Chemical	330
U S A	Hercules	2300
	BASF/Celion	1000
	AMOCO Performance Products	1000
	AKZO/Fortafil	1000
	Courtaulds-Grafil	950
	British Petroleum/Hitex	60
	Zoltek/Panex	250
U K	Courtaulds-Grafil	770
France	SOFICAR	770
Germany	AKZO	660

Pitch based		
Japan	Kureha Chemical	2000
U S A	AMOCO Performance Products	500
	Ashland Petroleum	300

Table 14

Fiber Manufacturers Grouping

Toray	AMOCO	SOFIC	5,070,000 lbs/year
Toho Rayon	BASF	AKZO	4,880,000
Hercules			2,300,000
Courtaulds			1,720,000

Table 15

World Consumption, PAN based Carbon Fibers

1000lbs/year

Year	1987		1988	
U S A	5000	(49%)	5800	(50%)
Europe	1720	(17%)	1980	(17%)
Japan	1870	(18%)	1940	(17%)
Far East, Others	1740	(17%)	2000	(17%)
Total	10330	(100%)	11720	(100%)

1987	Aerospace		Sports		Industrial	
U S A	3300	(65%)	700	(15%)	1000	(20%)
Europe	970	(56%)	330	(19%)	420	(24%)
Japan	90	(5%)	1430	(76%)	350	(19%)
Far East, Others	90	(5%)	1390	(80%)	260	(15%)
Total	4450	(43%)	3850	(38%)	2030	(19%)

Table 16

Japan-Carbon Fiber Industries

Manufacturer	Fibers	Prepregs	Composites
Toray Industries	—	—	—
Toho Rayon	—	—	—
Asahi Kasei CF	—	—	—
Mitsubishi Rayon	—	—	—
Kureha Chemical	—		—
Koa Oil	—		
Mitsubishi Chemical	—	—	
Kashima Oil	—		
Nippon Carbon	—		—
Sumitomo Chemical		—	
Yokohama Rubber		—	—
Arisawa		—	—
Aerospace Mfrs			—
Sporting Goods Mfrs			—
Industrial Goods Mfrs			—

Table 17

Agreement, Licensing and Sales

Licensing, PAN Precursor and Carbon Fiber Manufacturing

to UCC / AMOCO Performance Products

in 1978 Agreement, 1982 Start-up

on Thornel T300 High Performance Carbon Fiber

Cross-Licensing, Prepreg Resins

to Hexcel

in 1988

on Toughened Epoxy Resin 3900-2 of Toray and
Bismaleimid Resin F-655 of Hexcel

Sales Agreement, Carbon Fibers

with TOMAC
AMOCO Performance Products (under negotiation)

Table 18

Advanced Composites Manufacturer

Aircraft	MHI KHI FHI SMIC
Spacecraft	MELCO Toshiba NEC IHI MHI KHI FHI
Industrial	Sumitomo-denko Nitto-denko Hitachi-kasei
Sporting	Mizuno Daiwa-seiko Yamaha
Carbon/carbon	Nissan-motor

Table 19

Application

	Now			Future	
Aircraft	B767	F15J	T-4	B7J7	FSX
Spacecraft	Satellite		Rocket	Space plane	
Jet engine	V2500				
Energy				Fuel cell	Wind mill
Medical	X ray equipments				
Building	Concrete panel				
Sporting	Fishing rod Golf club Tennis racket Yacht	Boat	Wind surf	Bicycle	
	Racing car				

Table 20

R & D Activity

Min. Intern. Trade & Industry

Agency Industrial Sci. Tech. ———— Mechanical Eng. Lab.,Products Res. Lab., Chem. Lab.
Osaka Ind. Lab., Nagoya Ind. Lab., Kyushu Ind. Lab.

Min. Educ. Sci. Culture ———— Universities, Institutes, Colleges

Sci. Tech. Agency ———— National Aerospace Lab.

Defense Agency

National Space Develop. Agency
R & D Inst. Metals & Composites for Future Industries

Japan Soc. Composite Materials
Japan Soc. Mech. Engineers
Japan Soc. Aeronaut. Space Sci.
Japan Soc. Materials Sci.
Japan Soc. Fiber Sci. Technol.
Japan Soc. Polymer Sci.

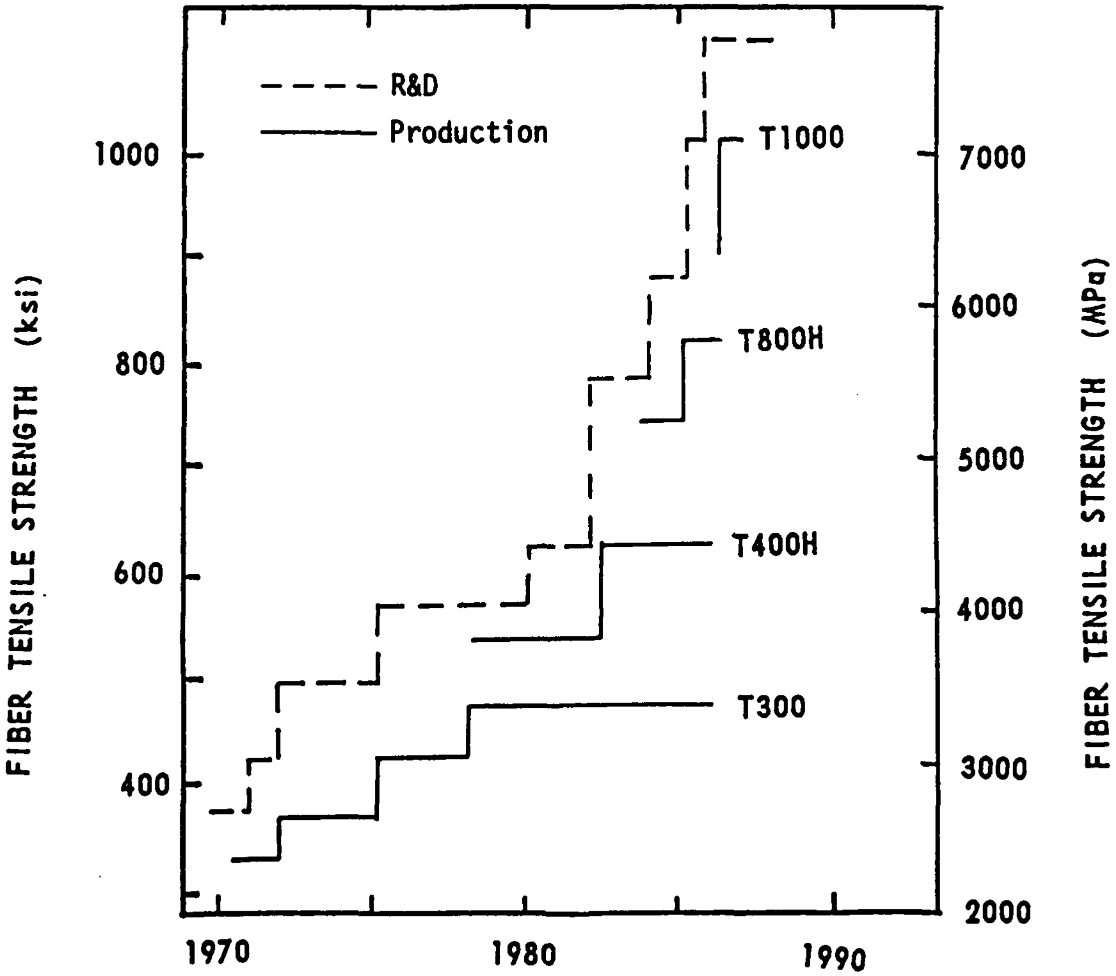

R&D
Production
T1000
T800H
T400H
T300
FIBER TENSILE STRENGTH (ksi)
1000
800
600
400
FIBER TENSILE STRENGTH (MPa)
7000
6000
5000
4000
3000
2000
1970
1980
1990

Figure 11

"Torayca" Carbon Fibers, Tensile Properties

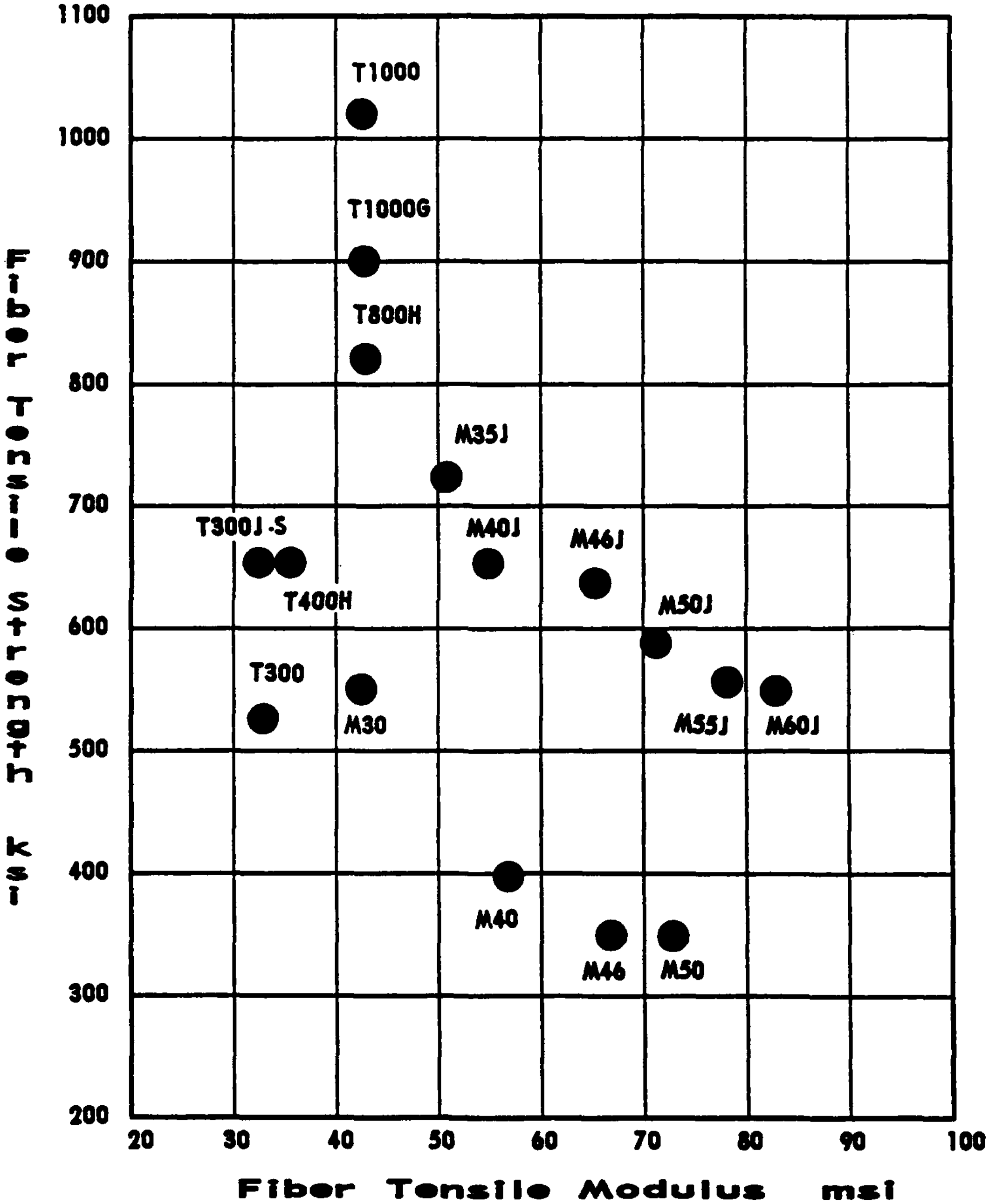


Figure 12

Carbon Fiber Manufacturing Process

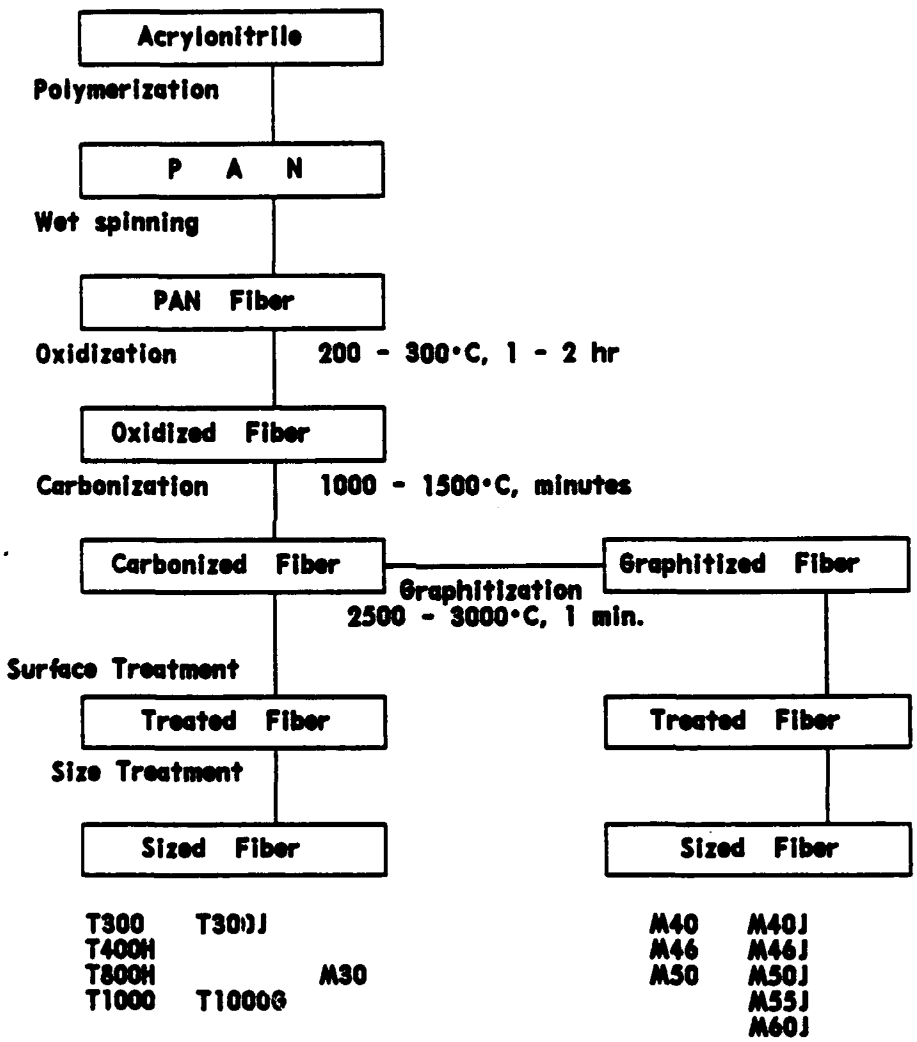
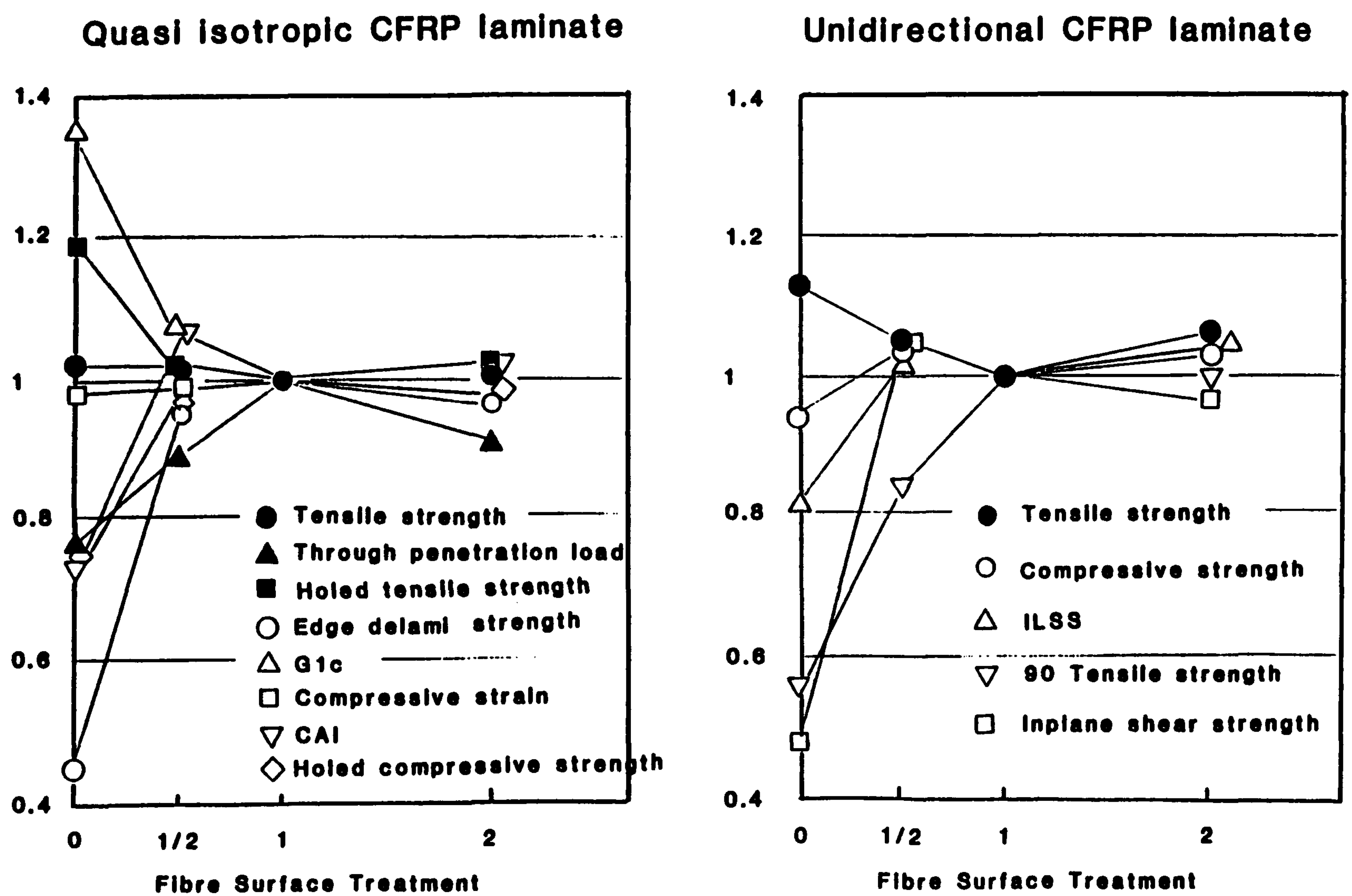


Figure 13



Quasi isotropic CFRP laminate
1.4
1.2
1
0.8
0.6
0.4
0
1/2
1
2
Fibre Surface Treatment
Tensile strength
Through penetration load
Holed tensile strength
Edge delami strength
G1c
Compressive strain
CAI
Holed compressive strength
Unidirectional CFRP laminate
1.4
1.2
1
0.8
0.6
0.4
0
1/2
1
2
Fibre Surface Treatment
Tensile strength
Compressive strength
ILSS
90 Tensile strength
Inplane shear strength

Figure 14

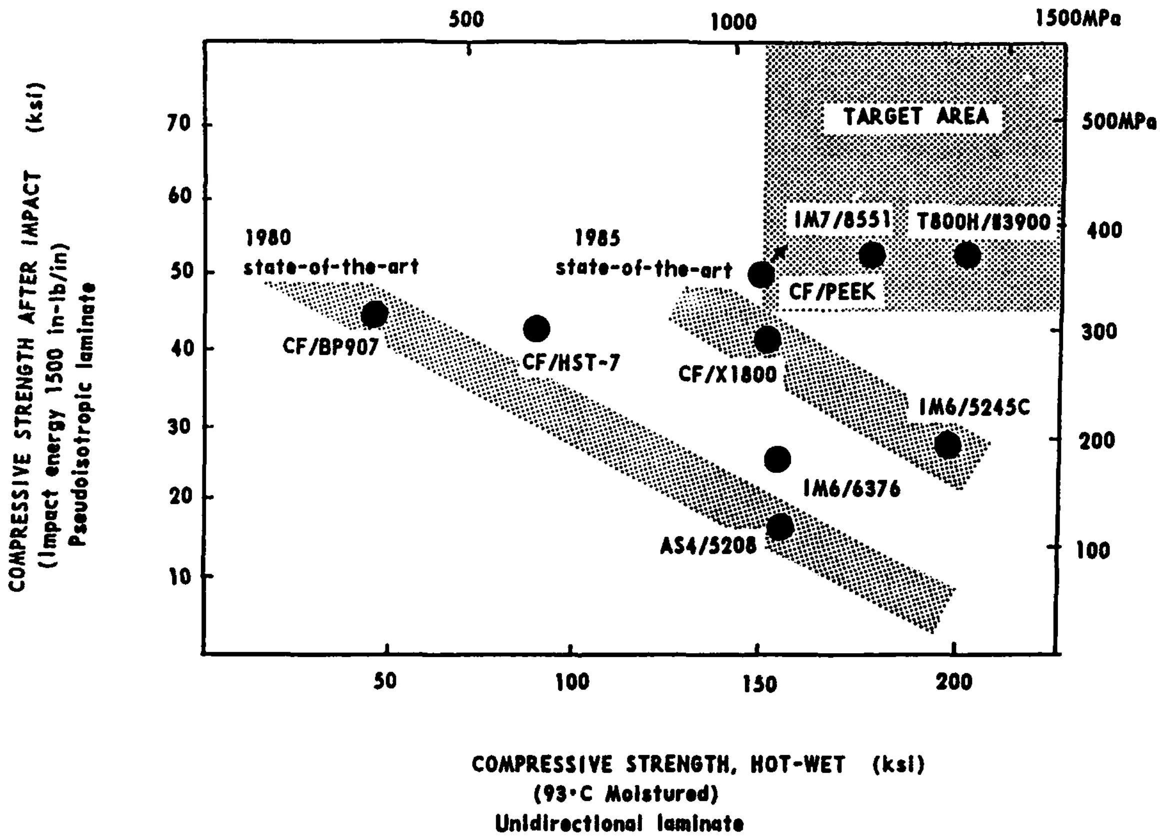

500
1000
1500MPa
500MPa
400
300
200
100
70
60
50
40
30
20
10
50
100
150
200
TARGET AREA
IM7/8551
T800H/#3900
1980
state-of-the-art
1985
state-of-the-art
CF/PEEK
CF/BP907
CF/HST-7
CF/X1800
IM6/5245C
IM6/6376
AS4/5208
COMPRESSIVE STRENGTH AFTER IMPACT (ksi)
(Impact energy 1500 in-lb/in)
Pseudoisotropic laminate
COMPRESSIVE STRENGTH, HOT-WET (ksi)
(93°C Moistured)
Unidirectional laminate

Figure 15

- Toray has no experience with ceramic matrix composites because they have found no demand.
- Toray started R&D in metal matrix composites in the early 1970s, but there has been no demand or market in Japan, so the effort is very modest.
- There is no current or planned effort in pitch-based carbon fibers; Toray had a pilot plant but closed it. The raw materials were cheap, but the processing was expensive for the properties Toray got. Quality control of the pitch was a problem.
- Kobe Steel, Nissan, and Nippon Carbon are working on C-C. Toray is interested in knowing how and why to make C-C.
- Toray had a modest effort on the development of Al_2O_3 ceramic fibers but stopped it for technical and market reasons.
- Toray is selling substantial amounts of C fibers, prepreg, and composites in Japan and fibers overseas.
- The prices for C fibers have been steady for several years, while the properties and quality have improved. R&D, qualification to specifications, and quality control are expensive--about one-third of fiber costs. Toray believes most or all companies producing C fibers are losing money; this is a factor causing numerous producers to sell their C fiber interests.
- Staff members reiterated their view that, at least at Toray, they are approaching theoretical limits for the fibers, and emphasis is needed on resins, interfaces, and automated processing. Toray has a large effort aimed at characterizing and tailoring fiber surfaces in order to control the interface.

2. Resins and Composites

At Toray, most thermoplastic composites are based on commodity resins: nylons, polypropylene, polyurethane, polyesters, ABS, and PPS. A few charts listing properties are included here (Tables 21, 22).

Toray produces several 250°F cure epoxies, especially for sporting goods, and a number of 350°F cure epoxies that range from first to third generation in performance. Toray's 3900 resin has been licensed to Hexcel; a composite data sheet is included here (Tables 23, 24, Fig. 16).

Toray is also increasingly interested in high-temperature composites such as bismaleimides, polyimides, and PPQs. Its work on NTPQ (Figs. 17, 18) was sponsored by MITI. As Japanese interests in aerospace grow, as exemplified by their plans for an

Table 21

TP－ACM

Thermoplastics-Advanced Composite Materials

Components:

- Fibre
  - CF "TORAYCA"
  - GF
  - AF "Kevlar"
  - UD (yarn)
  - Fabric
  - Chopped yarn
- Matrix
  - Polyamide
  - Polypropylene
  - Polyurethane
  - Polyester (PET, PBT)
  - ABS, PPS

Table 22

Properties of TP-ACM

Matrix: Nylon6

Fibre	CF "TORAYCA"		GF	
Fibre Configuration	Chopped Yarn	UD (Yarn)	Chopped Yarn	Short Fibre
Fibre Length (mm)	25		25	0.3
Fibre Weight Fraction (%)	50	70	60	45
Fibre Volume Fraction (%)	40	60	40	27
Tensile Strength (kgf/mm^2)	25	170	20	20
Modulus (kgf/mm^2)	2700	13300	1600	1200
Flexural Strength (kgf/mm^2)	50	190	40	30
Modulus (kgf/mm^2)	2500	13200	1400	1200
Izod Impact Strength notched (kgf·cm/cm)	20		120	15
Heat Distortion Temp. (℃)	215	215	215	205
Density	1.38	1.53	1.70	1.48

Table 23

RESIN	CURE TEMP.	CODE No.
EPOXY	250°F	#2500, #2571, #2580
	350°F	#3601, #3620, #3631 #3900-2, #3910
BMI	350°F	Under Development
POLYIMIDE	600°F	Under Development
PPQ	500°F	Under Development
	600°F	Under Development

Table 24

Mechanical Properties of T800H/3900-2 Composites

Properties		Unit	Temp.	T800H/#3900-2[1]
Tensile	Strength	MPa(ksi)	RT	2690(391)
	Modulus	GPa(msi)	RT	157(22.8)
	Strain	%	RT	1.68
0° Compressive Strength		MPa(ksi)	RT	1690(245)
			82℃	1450(211)
			82℃-Wet[2]	1320(191)
			RT[3]	1380(200)
Compressive Interlaminar Shear Strength		MPa(ksi)	RT	77.2(11.2)
			82℃	55.1(8.0)
			RT[3]	77.8(11.3)
Open Hole Tensile Strength		MPa(ksi)	RT	451(65.4)
Open Hole Compressive Strength		MPa(ksi)	RT	316(45.8)
			82℃	274(39.7)
			82℃-Wet[2]	304(44.1)
Compressive Strength after Impact Impact Level 67 J/m		MPa(ksi)	RT	367(53.3)
			82℃-Wet[2]	349(50.6)
			RT[3]	337(48.9)

1) Fibre areal weight of prepreg:145 g/m²
2) Immersed 2 weeks in 71℃(160°F) water
3) Mecanical life test:Cure after exposure at RT for 1000 hr

Properties of TORAYCA Composites

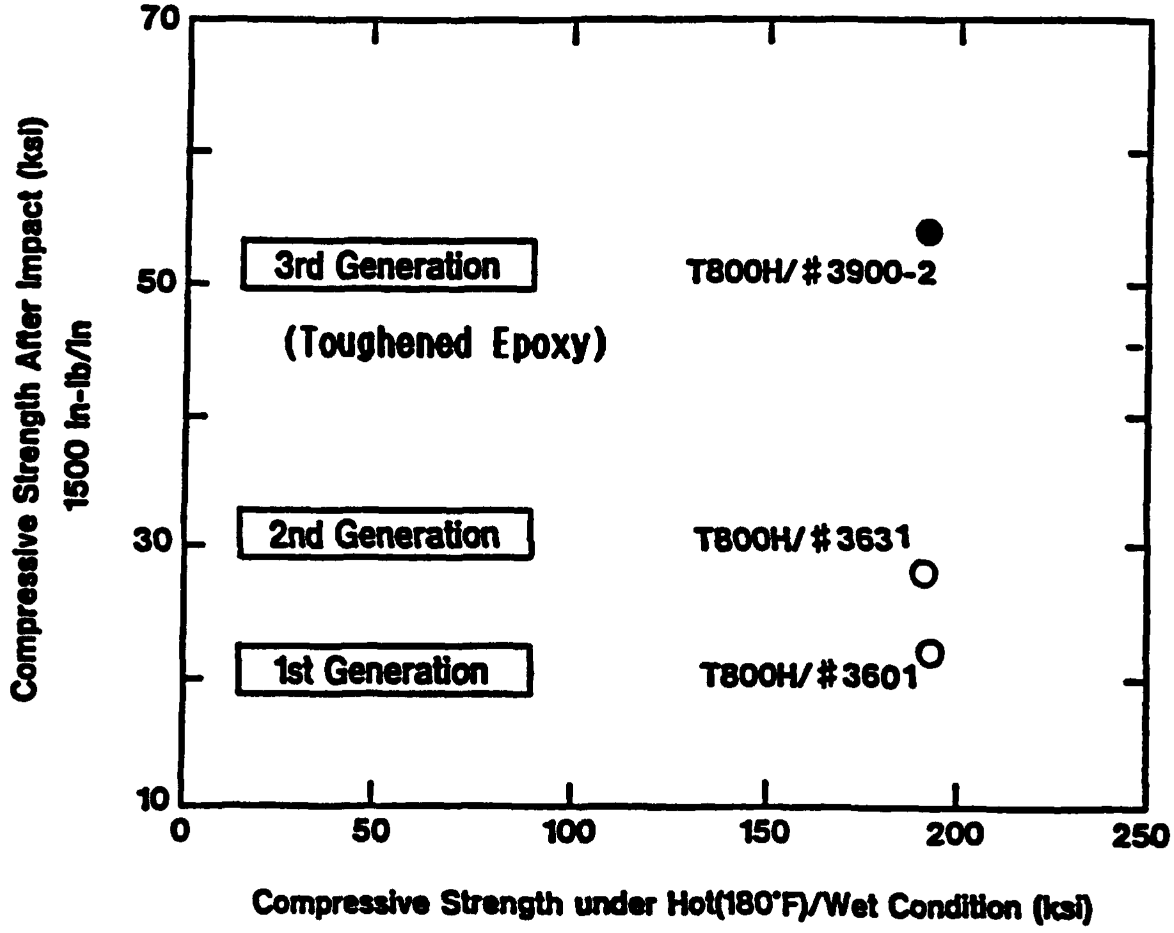


Figure 16

ADDITION CURABLE NTPQ

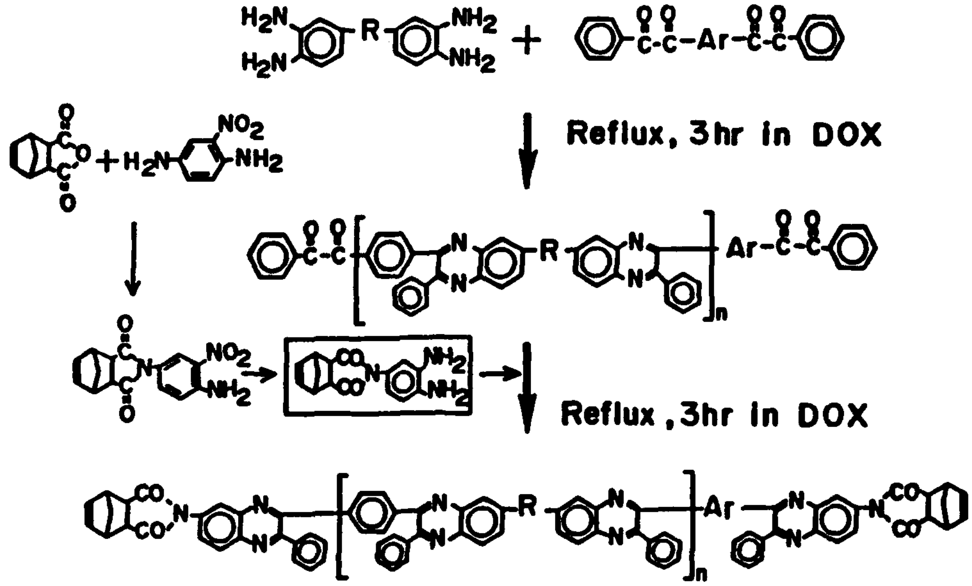
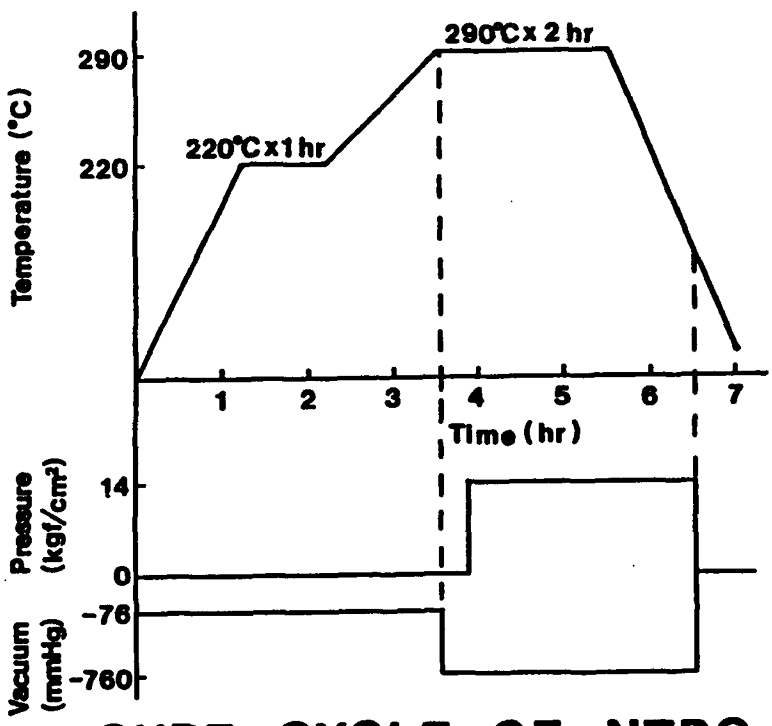


Figure 17



CURE CYCLE OF NTPQ

Figure 18

orbiting plane, HOPE, in the 1990s, and as organized by the new MITI high-temperature/high-performance materials program initiated in April 1989, all Japanese materials suppliers appear to see reasons to be interested in higher temperature materials.

The visit to Toray concluded with tours of the fibers analysis laboratory at the Toray Research Center and the Toray Prepreg Production Line. Both tours were impressive for the cleanliness of the operations and the quality of the equipment.

E. TOYOTA CORPORATE RESEARCH AND DEVELOPMENT LABORATORY

On February 6, 1989, the TAT visited the Toyota Corporate Research and Development Laboratory (CRDL) in Nagakute, Aichi, near Nagoya. The Toyota Group consists of many companies, among them the Toyota Motor Corporation, the Aichi Steel Works, the Toyoda Automatic Loom Works, the Toyoda Machine Works, the Aisin Seiki Company, and the Toyoda Spinning and Weaving Company. The Corporate R&D Laboratory is a think tank intended to assist all of the Toyota Group. A breakdown of the CRDL organization, research fields, and recent awards is included here (Tables 25, 26).

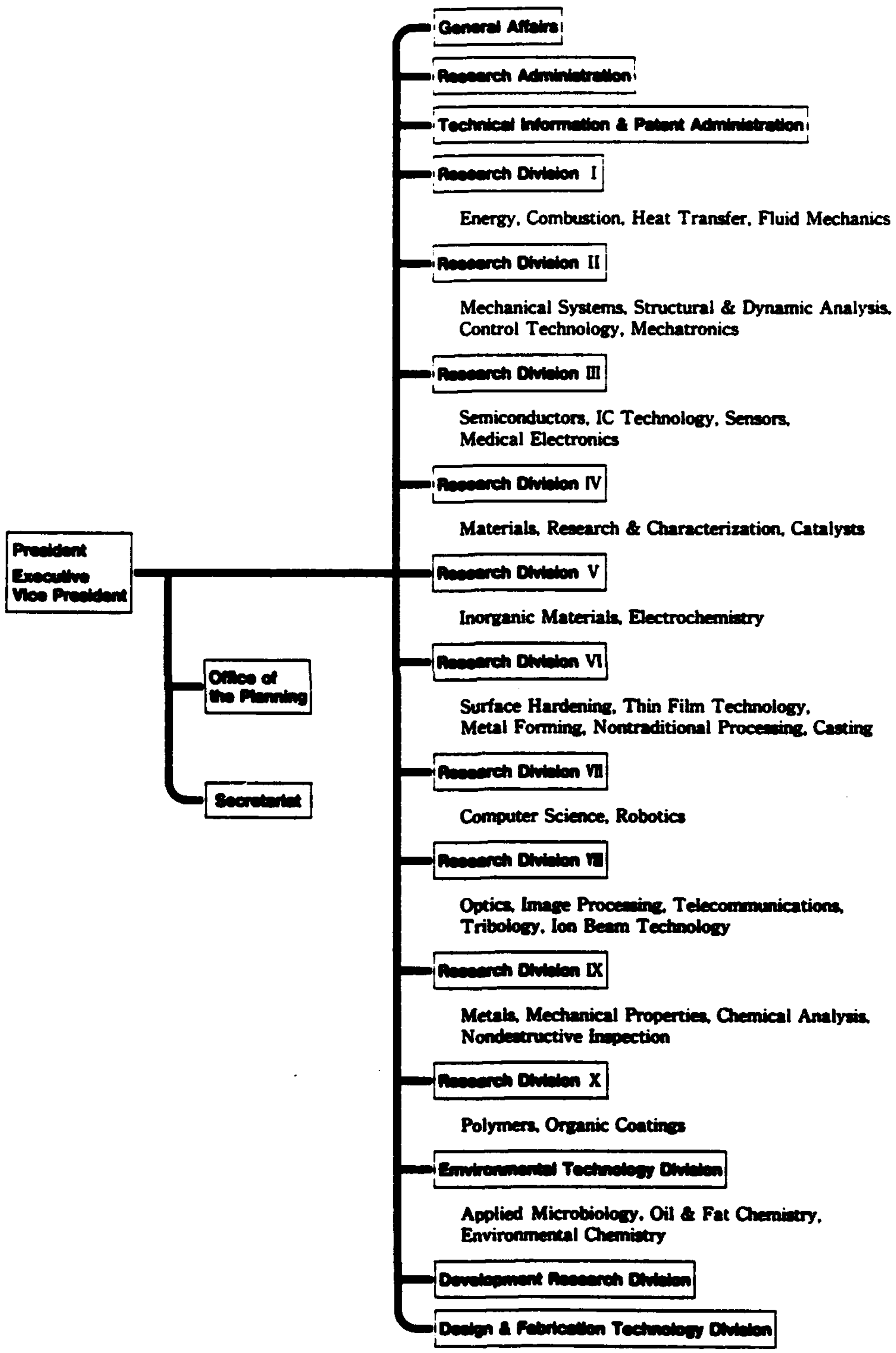
1. Metal Matrix Composites

After an introductory video, the TAT was given an extensive presentation on Fiber/Dispersoid Hybrid Fiber-Reinforced Metal Matrix Composites, a Toyota CRDL development (described to the TAT at Ube Industries) for which Toyota CRDL received a 1988 IR-100 award. A four-page handout is included here (Table 27, Figs. 19-21). Basically, Toyota CRDL says that earlier MMC processing problems, such as fiber degradation due to reactions between the fiber and the matrix, development of brittle second phases, fiber-to-fiber contacts providing preferred crack propagation paths, and control of fiber volume fraction, are all obviated by incorporating whiskers or particulates between the fibers to prevent fiber-to-fiber contact. It was not clear how all these problems were solved by one simple step, but CRDL spokesmen mentioned elsewhere that reactivity between the matrix and the fiber is due more to the alloy than to Nicalon or Tyranno fibers. (Ni additions in the alloy reduce reactivity.) Toyota CRDL claimed the ability to mass-produce complex shapes to near net shape by squeeze-casting, using the Hybrid FRM process. Typically, CRDL would use an Al 4.5 Cu alloy reinforced with 15 μ diameter Nicalon fibers from Nippon Carbon Company or Tyranno fibers from Ube. Whiskers used to separate the fibers could be 0.2 to 0.5 μ in diameter by 50 to 200 μ long from Tokai Carbon or 0.05 to 0.2 μ by 10 to 40 μ SiC from Tateho. Particulate separators of about 0.28 μ come from Ibiden Betarunden.

A discussion of the MMC used in the pistons of Toyota diesel engines revealed that Toyota Motor Company, not CRDL, has worked on MMCs for 25 years, and its staff did the R&D for the current application. CRDL has worked on MMCs for the past 10 years and is focusing on continuous-fiber MMCs.

Table 25

ORGANIZATION & RESEACH FIELDS



(Employees : ca. 870) 1989.2

Table 26

LIST of AWARDS (from 1986)

OVERSEAS AWARDS

1986 **Fédération Internationale des Sociétés Ingenieurs des Techniques de l'Automobile, Overall Economy Prize**
Measurement and Simulation of Air Motion in a Re-entrant Combustion Bowl for a DI Diesel Engine
I・R 100 Prize
Noise Level Prediction Computer Code, "ACOUST/BOOM"
I・R 100 Prize
Wheel Torque Measuring System, "WTMS"

1987 **I・R 100 Prize**
Co/Spinel Catalyst for Producing Carburized Gas
I・R 100 Prize
Engine Combustion Simulation System (ECOS)

1988 **ICLASS-88, YASUSHI TANASAWA Award**
Injection Valve of Swirl Flow Type Used for Electronic Fuel Injection
R&D 100
Thin Film Limiting Current Oxygen Sensor
R&D 100
Fiber/Dispersoid Hybrid Fiber-Reinforced Metal Matrix Process

DOMESTIC AWARDS

1986 **Society of Automotive Engineers of Japan, Thesis Prize**
Photographic Observation of Knock with a Rapid Compression and Expansion Machine

1987 **Japan Society of Color Material Society, Thesis Prize**
Study on Paint Atomization and Film Formation
Japan Society of Mechanical Engineers, Technology Prize
Development of Styling CAD System
Commendation by Minister of State for Science and Technology
Development of Ceramic Coating by Diffusion Process
Research Prize of the Ceramic Society of Japan
Study on Reliabilities of Ceramics
The Japan Institute of Metals, The Best Year's Papers Award
Ion Implantation Induced Phase Transformation in Fully Stabilized Zirconia

1988 **Japan Society of Medical Electronics and Biological Engineering, The Science News Award**
Monolithic Pressure-Flow Sensor
Japan Society of Lubrication Engineers, Best Paper Award
Corrosive Wear of Iron Castings in Acids
Japan Society for Technology of Plasticity Awards Winners, JSTP Paper Prize
Effects of Rolling Conditions on Accuracy of Hot Form-Rolled Helical Gears
Tokai Chemical Industry Association, Best Research Prize
Study of Three-Way Catalysts for Automotive Emission Control
Japan Fine Ceramics Association, Technical Award
Research on Solid Solutions of Nitride Fine Ceramics
Japan Society of Coating Technology, Technical Award
Quantitative Evaluation of Coating Appearance Qualities
Japan Foundrymen's Society, Kusaka Promotion Prize
Clarification of Mechanism of Solidification Process Using Instrumental Casting Technology
Japan Foundrymen's Society, Kobayashi Prize for Best Paper
Crystallization of AlFeSi Compound in Chinese Script Form in Melt-Superheated Al-Si Alloy Castings
Textile Machinery Society of Japan, Best Paper Prize
Jets Injected into Air Guides of an Air Jet Loom

Table 27

Fiber/Dispersoid Hybrid

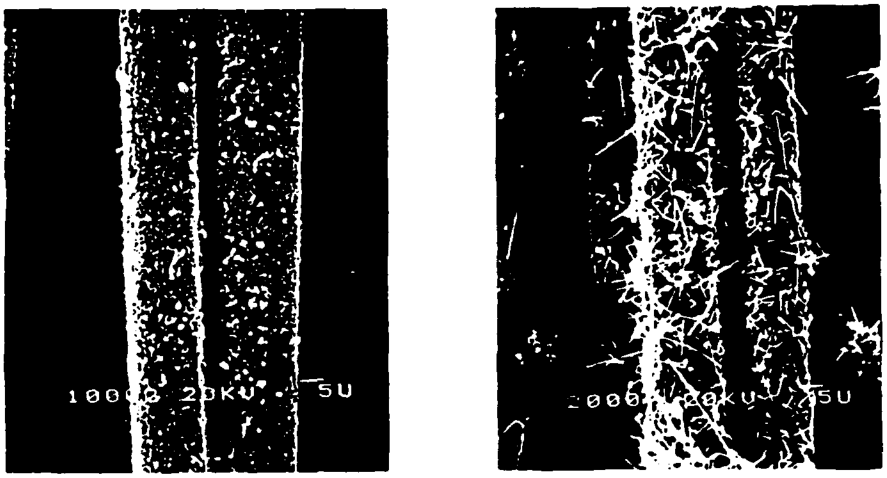
Fiber-Reinforced Metal Matrix Composites

Features

Fine whiskers and/or particulates are distributed among the continuous fibers.

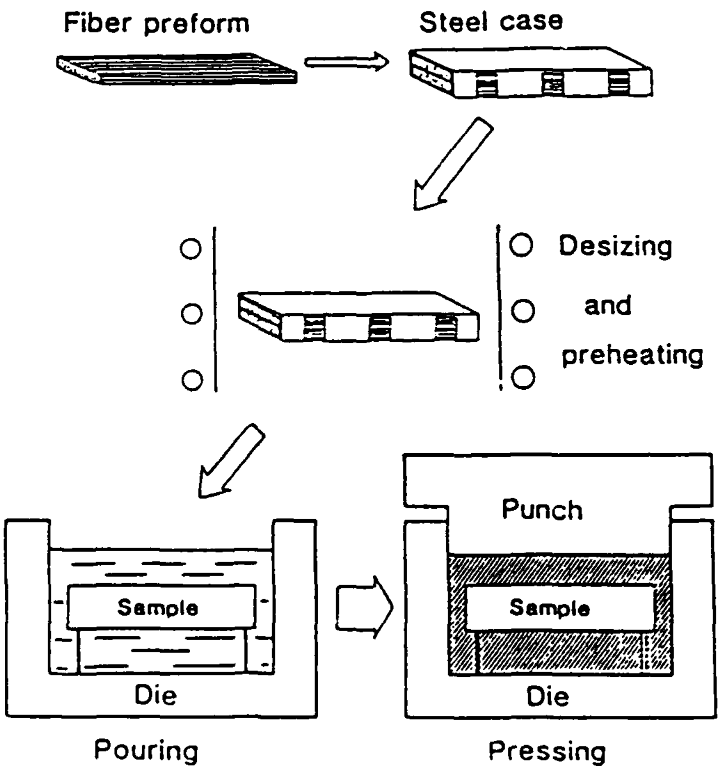
1. Uniform distribution of continuous fibers
2. Improvement of solidification mechanism
3. Inhibition of degradation of fiber caused by interfacial reaction
4. Volume fraction control of fine continuous fibers

TOYOTA CRDL



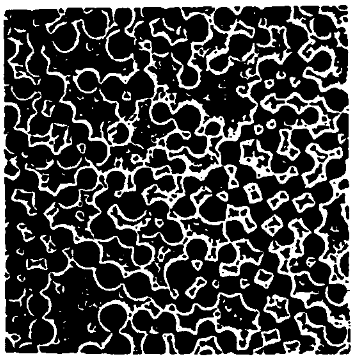
SiC fiber + SiC particultes   SiC fiber + SiC whiskers

Hybrid fiber

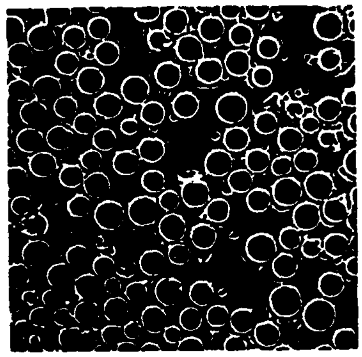


FRM fabrication by a squeeze casting process

Figure 19

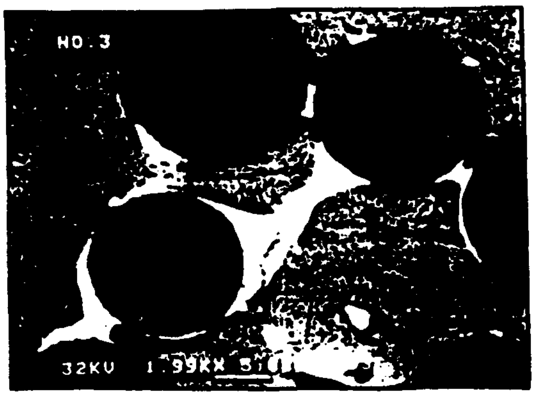


conventional composite

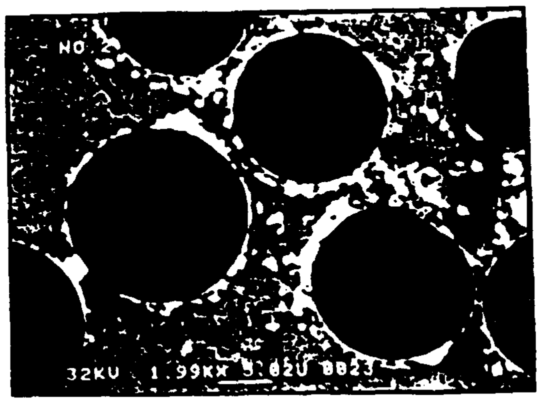


particulate hybrid

Transverse cross sections of composites



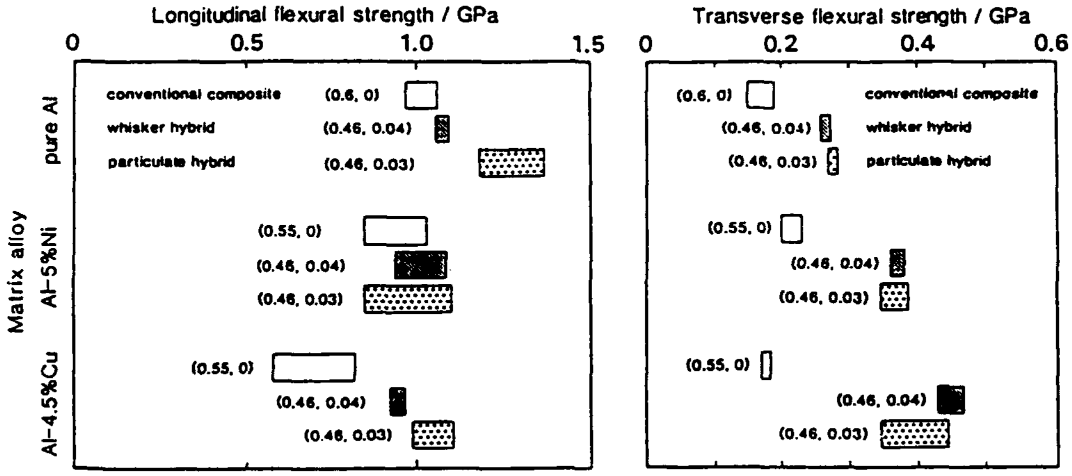
conventional composite



particulate hybrid

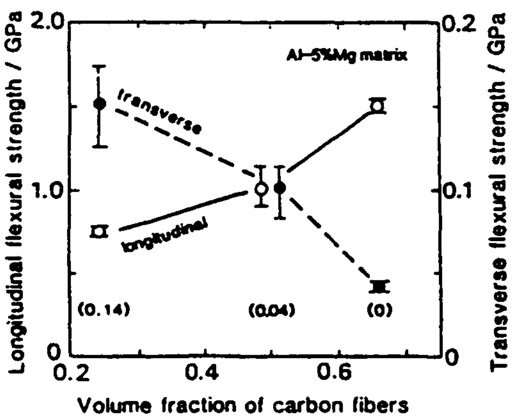
Back-scattered electron images of Al-Cu matrix composites.
Bright images indicate copper segregation.

Figure 20



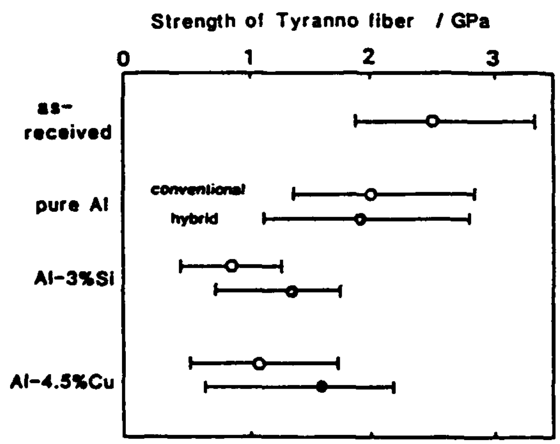
Mechanical properties of silicon-carbide fiber composites.

The number in the parentheses are represent the volume fraction of fiber and dispersoid.



Volume fraction control

The numbers in the parentheses represent the volume fraction of whiskers.



Control of interfacial reaction between fiber and matrix metal.

Figure 21

2. Ceramics and Ceramic Matrix Composites

The Toyota speaker began this presentation with the question: Since the CMC papers presented at the Japanese Ceramic Society numbered 6 in 1986, 12 in 1987, and 9 in 1988, while 74 were presented at American Ceramic Society meetings in 1986 and 70 in 1988, why visit Toyota CRDL? Later, when the results of a burst test on a ceramic turbine wheel were shown, a speaker observed that such work was done in the United States 6 years ago, so the Japanese feel they are 6 years behind the United States in that area.

CRDL has worked on whisker-reinforced CMCs. Spokesmen described some injection-molded silicon nitride reinforced with SiC whiskers where the whiskers were aligned in the injection molding process, resulting in 8 percent shrinkage in length while the width and thickness had 20 percent shrinkage. Overall, they betrayed considerable ambivalence about whisker-reinforced ceramics, claiming they are too difficult and expensive to process when compared with monolithic ceramics. At two different times, they stated that they considered monolithic ceramics more attractive than whisker-reinforced CMCs, since they could achieve similar properties at lower cost. They described a hot-pressed silicon nitride made with Ube powder that had a room-temperature four-point bend strength of about 180 KSI, and a strength of 140 KSI at 1300°C. K_{IC} was 6.5 and grain size was mostly round at 0.3 to 0.5 μ. They professed not to know why the material is so good and said they would report publicly on it at the European Ceramic Conference in June 1989. Though data was scarce at the time of the TAT visit, they expected appreciable high-temperature oxidation resistance.

During subsequent discussions, CRDL personnel reiterated the preference for monolithic ceramics over whisker-reinforced CMCs and expressed curiosity at U.S. emphasis on reinforcement. However, they did say they want to do continuous-fiber-reinforced CMC work as soon as they find staff available.

The visit to CRDL concluded with a tour of the laboratory (very impressive equipment and facilities) and a visit to the exhibit room where about 40 displays illustrated CRDL successes. The most interesting display was an exhibit of ceramic ball bearings (balls and races) that had been used in hot corrosive environments. CRDL says sintered and HIP'd silicon nitride bearings are better than hot-pressed.

F. ISHIKAWAJIMA-HARIMA HEAVY INDUSTRIES COMPANY, LTD. (IHI)

The TAT visited Ishikawajima-harima Heavy Industries Company, Ltd. (IHI) on February 7, 1989. Initial briefings took place in the Hotel Marunouchi in Ohtemachi (Tokyo) near the IHI Headquarters Office. In the afternoon, the TAT toured the IHI Research Institute in Toyosu (also Tokyo, on land reclaimed from Tokyo Bay).

IHI, founded in 1853, has grown to 16,500 employees and annual sales of ¥770 billion. The main products are: aeroengines, space-related equipment, and gas turbines (14 percent); land-based machinery and plants (69 percent); and ships (17 percent). IHI has produced numerous turboprop, turbofan, turbojet, and turboshaft engines under license from General Electric, Pratt & Whitney, and Rolls-Royce, including the F100, J79, and T700. IHI is currently a part of the consortium building V2500 turbofan engines.

IHI produces boilers and thermal power plants; reactor pressure vessels, containment vessels, heat exchangers, and piping for nuclear power plants; hydroelectric dams and power facilities; and gas-turbine- and diesel-powered generators. IHI builds oil refineries, petrochemical plants, and desalination plants and a wide range of materials handling and industrial robot equipment. IHI builds bridges, tunneling equipment, and air pollution controls. IHI is also a major shipbuilder and contractor of offshore drilling rigs.

In recent years, IHI has been an active participant with the National Space Development Agency of Japan (NASDA), the National Aerospace Laboratory (NAL), and the Institute of Space and Aeronautical Science (ISAS) in various space development projects, including gas jet systems for attitude control, rocket engines using storable propellants, cryogenic propulsion systems using liquid oxygen and liquid hydrogen, microgravity processing, and ground test facilities. IHI produced several components of the H-1 Launch Vehicle.

IHI has an extensive and broad-based R&D program. IHI has Japan's largest research wind tunnel (test section 6 m × 3 m × 24 m), a 3D seismic simulator with a 4.5 m square vibration table of 35 ton capacity, and a huge tank to test the seakeeping and maneuvering ability of ships and the yawing of barges. IHI is very active in the development and application of laser and beam technology; in the research, development, and manufacture of superconductive magnets; in the development of ultrahigh-vacuum facilities; in fuel cells, biotechnology, and the application of CAD/CAM/CAE systems. To support and advance those technologies, the development and application of new

engineering materials tops the IHI priority list of R&D activities. The technology tree in Fig. 22 illustrates IHI's dedication. Superalloys, fine ceramics, polymer and metal matrix composites, and thin films are getting current emphasis so that IHI can supply its own advanced materials when there is no suitable supplier; precision casting is a prominent example.

1. Metal Matrix Composites

IHI has used many fibers in MMCs, including Textron's SCS-2 and -6 SiC fibers, Ube's Tyranno, Nippon Carbon's Nicalon, and ion-plated or CVD filament preforms of C. As shown in Fig. 23, IHI filament winds onto a drum, using low-pressure plasma spraying of Al or superalloy powders or adding a metal foil to get a tape that can be laid up and vacuum hot-pressed at up to 1250°C. Figure 24 provides some information on SiC/Al. Figure 25 provides information on SiC/Hybrids where ion plated carbon fibers are laid up parallel or perpendicular to the SiC fibers, and Table 28 provides data on SiC/Ti.

Figure 26 illustrates the process for making FeCrAlY and Inco 900 series superalloys reinforced with thoriated tungsten fibers. Figure 27 illustrates some processing problems. In response to questions about diffusion barrier coatings, IHI said some information would be released at ICCM-6. Table 29 gives limited data, mostly single points for fiber-reinforced superalloys.

2. Superalloys

IHI is very active in superalloys and provided an excellent superalloy presentation, which will not be described here except to note areas of activity and status. IHI has completed development work on columnar grained blades, terminated its efforts on eutectic composite blades, and nearly finished development of single-crystal blades. It is continuing work on oxide dispersion strengthened (ODS) alloys and is doing feasibility studies on rapid solidification rate (RSR) through melt spinning, gradient effects on Ni-Ni_3Al, and processing, microstructure, and deformation studies of Nb intermetallics.

3. Ceramics and Ceramic Matrix Composites

IHI has a very substantial ceramics program, involving part-time efforts of 70 to 100 people. Its goals are not to supply ceramic materials, nor necessarily to achieve high performance. Its reasons are twofold: (1) Much of IHI's products and efforts require or

New Applications of New Material Technology to IHI Products
材料技術によるIHIの新事業・新製品展開
(Relevance Tree)
(関連樹木図)

S61.2.20
技企部

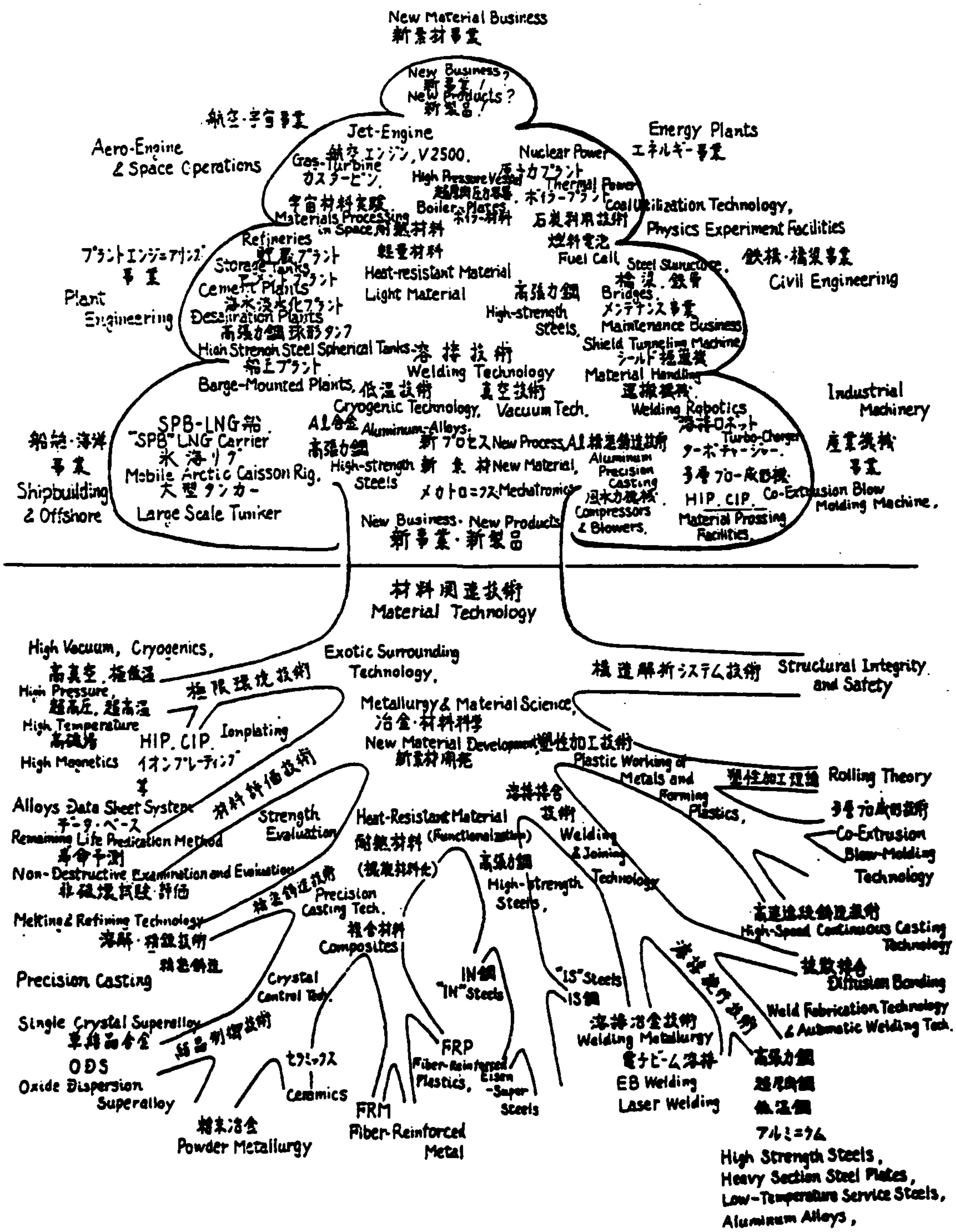


Figure 22

A. PROCESSES AND FACILITIES

1. PROCESS FLOW OF MMC

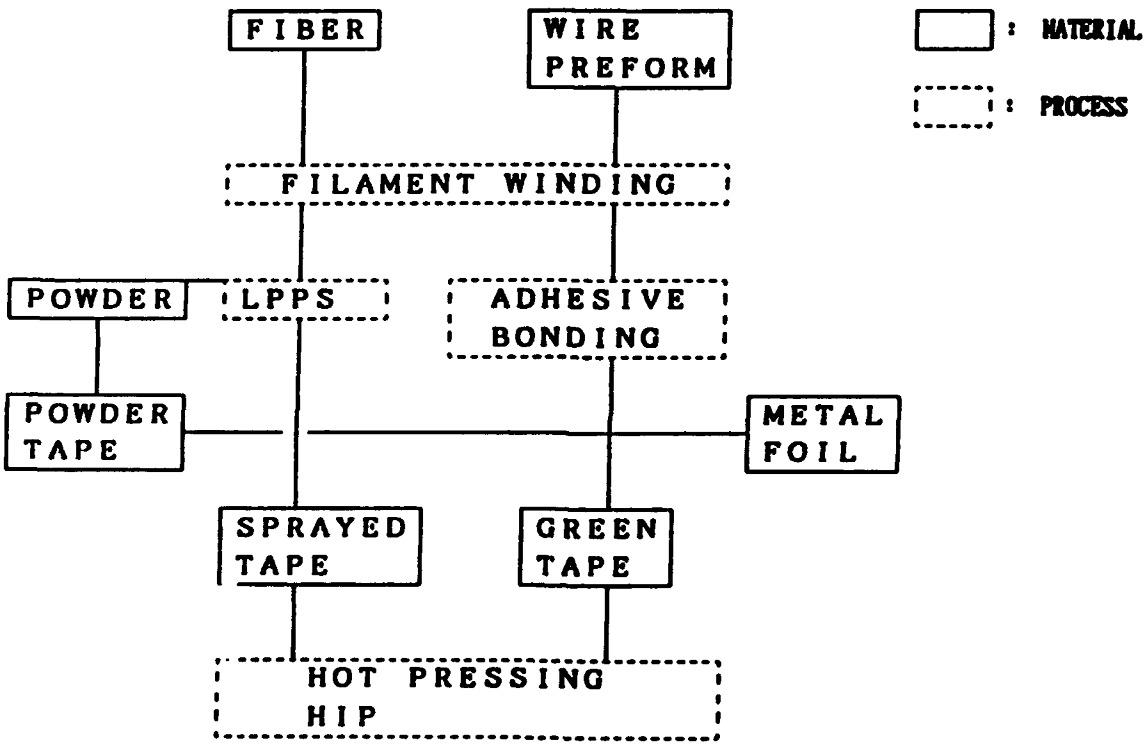


Figure 23

B. MATERIALS R&D

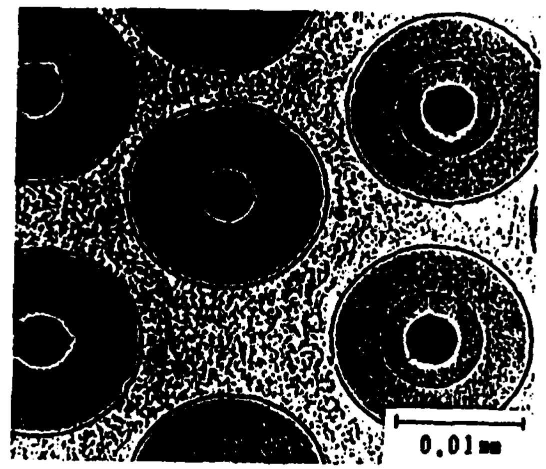
1. SiC(CVD) FIBER REINFORCED ALUMINUM MATRIX COMPOSITES

1・1 IMPROVEMENT OF MATRIX ALLOYS

MECHANICAL PROPERTIES

TENSILE STRENGTH

	RT	573K	723K
0°	1833 MPa	1679 MPa	1490 MPa
90°	49 MPa	52 MPa	44 MPa



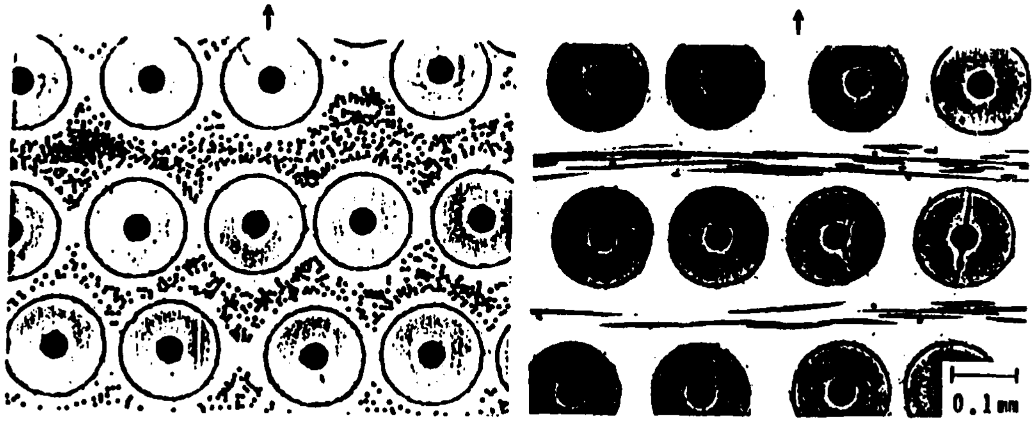
MATRIX : Al-8Fe-4Ce
Al-4Ti
Al-8Cr-1Fe

Figure 24

1.2 SiC/HYBRID

MECHANICAL PROPERTIES

		0/0		0/90	
		RT	723K	RT	723K
TENSILE STRENGTH (MPa)	0°	1800	1500	1200	1000
	90°	40	—	170	140
RUPTURE ELONGATION (%)	0°	0.90	—	1.0	—
	90°	0.05	—	1.20	—



A) 0.43SiCF/0.11CF (0/0) B) 0.34SiCF/0.1CF (0/90)

Figure 25

Table 28

2 SiC(CVD) FIBER REINFORCED TITANIUM MATRIX COMPOSITE

THE MECHANICAL PROPERTIES AT ROOM TEMP.
OF SiC(CVD)/Ti-15-3-3-3 (Vf=0.35)

PROPERTIES	STRENGTH (MPa)	YOUNG'S MODULUS (GPa)	RUPTURE STRAIN (%)
0°, TENSILE	1904 (1817-2044)	191 (187-194)	1.06 (0.97-1.17)
90°, TENSILE	550 (410-601)	134 (130-138)	0.99 (0.58-1.22)
0°, COMPRESSIVE	3370 (2950-4104)	190 (181-193)	2.06 (1.66-2.77)

N=10, AVE.(MAX.-MIN.)

A. PROCESSES AND FACILITIES

1. PROCESS FLOW OF FRS

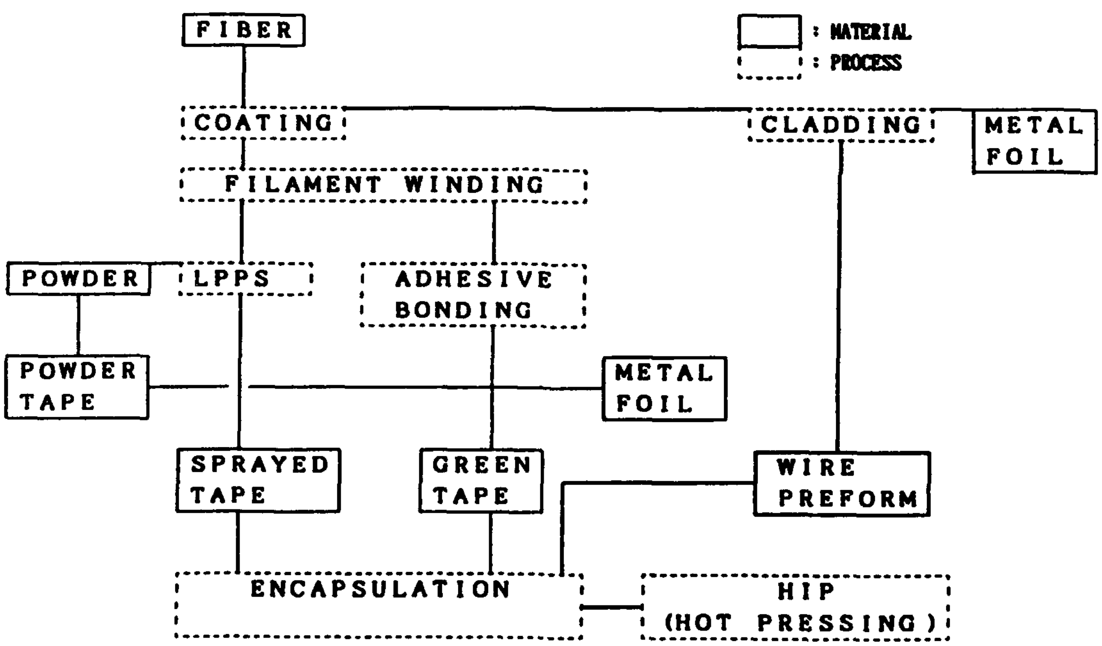
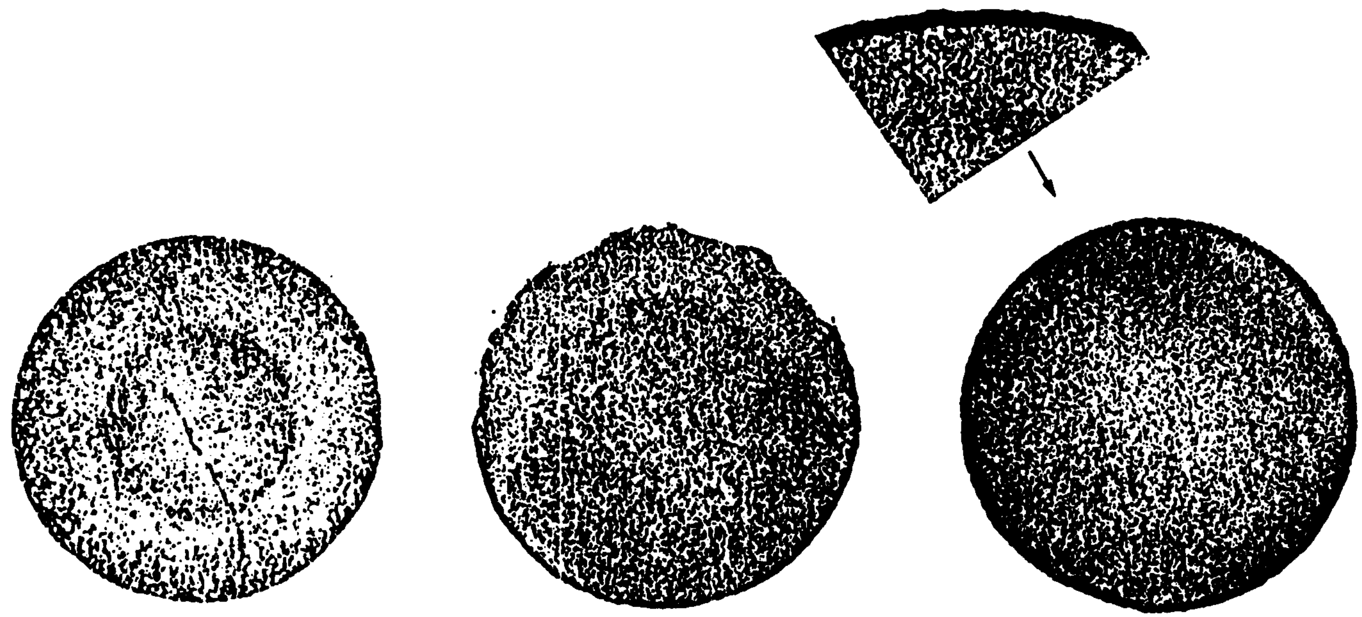


Figure 26

2. R&D OF THE DIFFUSION BARRIER COATING



A) RECRYSTALLIZED FIBER

B) RECRYSTALLIZED FIBER UNSUCEEDED COATING

C) SOUND FIBER WITH EFFECTIVE COATING

FIBER/MATRIX COMPATIBILITY AND THE EFFECT OF THE BARRIER COATING (1150℃ × 24 Hr)

Figure 27

Table 29

3. EVALUATION OF MECHANICAL AND PHYSICAL PROPERTIES

3. 1 TENSILE

	TENSILE STRENGTH
RT	980 MPa
700℃	620 MPa

3. 2 CREEP RUPTURE STRENGTH

TEMP.	STRESS	LIFE
900℃	280 MPa	480 Hr
1093℃	150 MPa	50 Hr

Vf=0. 26~0. 30

would benefit from ceramic components, so IHI wants to be able to make them if it cannot buy them, and (2) IHI designs and builds much process equipment, and it needs to do research on fabrication processes to support that effort.

Table 30 lists areas of application for ceramics, and Fig. 28 is a flow sheet of ceramic processing. Figure 29 shows two advanced applications.

IHI has done a little research on SiC whiskers in Si_3N_4, SiC, and PSZ matrices but has found no significant improvement in properties. Although the whiskers increased costs, the real cost increase came in processing. Work in continuous-fiber-reinforced CMCs has been negligible. No fiber was noted to be good above 1200°C.

4. Laboratory Tour

The IHI Laboratory was established in 1937. The laboratory in Tokyo focuses on materials and small hardware, and the laboratory in Yokohama on large-scale projects. The Tokyo laboratory does very much fatigue and environmental testing; space research including acoustics and electromagnetic levitation; combustion research using heavy oil, coal oil, pulverized coal, and natural gas; cryogenic studies; research on molten carbonate fuel cells; wind-tunnel testing of a 3D suspension bridge; studies of counterrotating marine propellers; and biotechnology research.

On our tour, we saw very much ceramic activity, ranging from slip casting to HIPing and considerable superalloy processing and fabrication. Most impressive was a display of turbomachinery--a veritable department store of turbos large and small, including many used in auto racing. It was said that processing accounts for one-third of the rotor cost and inspection for another third.

During concluding discussions, IHI personnel again mentioned our interest in CMCs, noting that CMC applications in man-rated jet engines are at least 15 to 20 years away and MMC applications about 10 years away. But they asked lots of questions about Lanxide, NASP, and work on the oxidation resistance of C-C. IHI plans to initiate R&D in C-C as part of the new MITI program.

Table 30

Major Areas of Applications of Engineering Ceramics

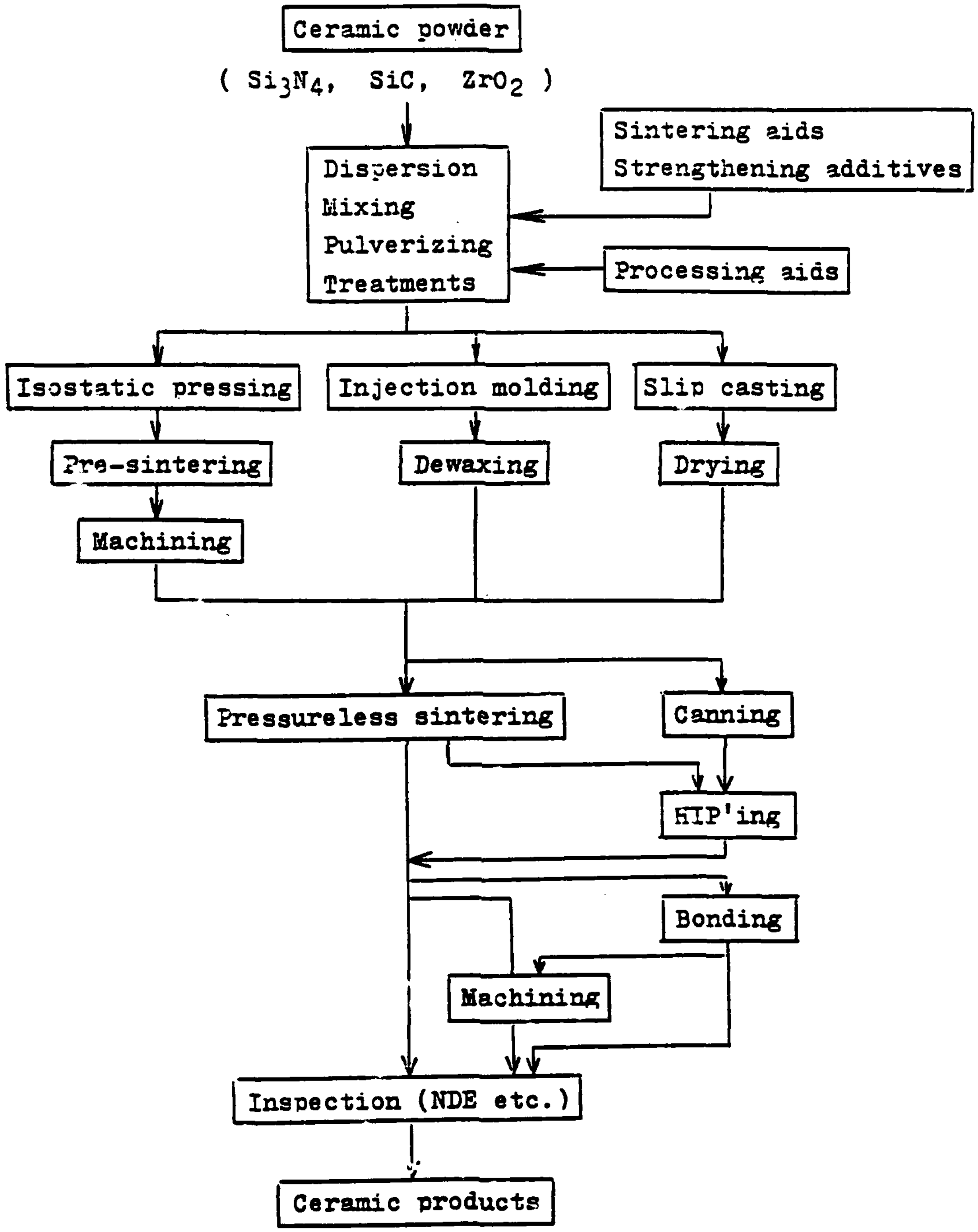
Chemical Engineering:
Catalysts, Sensors,
Corrosive & erosive structural components

Metallurgy & Materials Engineering:
Casting components,
Precision casting molds & cores,
High temperature structural components

Tribological & Precision Machinery:
Bearings, Valves, Frictional components

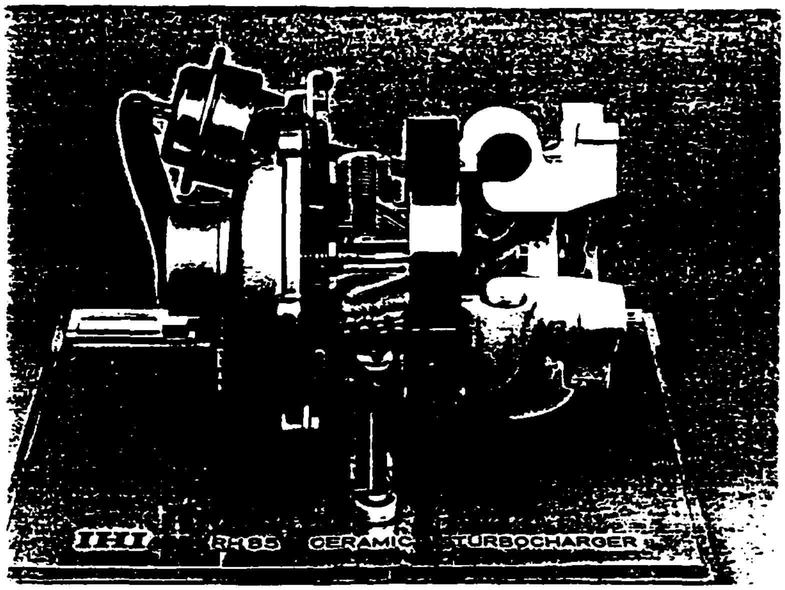
Mechanical & Transportation Engineering:
Turbomachinery components,
Diesel engine components

Energy Engineering:
Combustion chamber components,
Heat exchenger components,
High temperature structural components

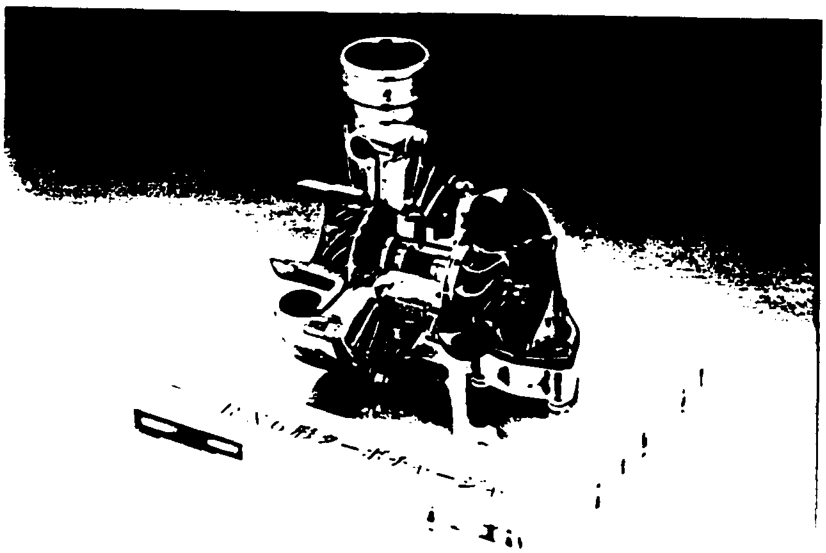


Flow Sheet of Ceramic Processing

Figure 28



TURBOCHARGER WITH A CERAMIC ROTOR AND A CERAMIC HOUSING



CERAMIC TURBOCHARGER FOR A RACING CAR

Figure 29

G. MITSUBISHI ELECTRIC COMPANY (MELCO)

On February 8, 1989, the TAT visited the Sagami Works of Mitsubishi Electric Company (MELCO) in Sagamihara near Tokyo.

MELCO was established in 1921. It has grown to be a leader in electronic and electrical equipment and includes 11 laboratories, 37 works and factories, and more than 75,000 employees. It is a major producer of electronic devices, communications and transportation equipment, building and industrial equipment, and audiovisual and home electronic equipment. It has led the Japanese space industry since its inception, acting as a prime contractor for 10 of the 19 satellites produced for the National Space Development Agency (NASDA).

MELCO spends 8 percent of its total annual sales on R&D. Roughly $800 million goes for basic research and exploratory development for defense purposes. The Japan Defense Agency benefits from but does not support this effort. MELCO claims other Japanese companies do likewise.

The Central Research Laboratory for MELCO is in Amagasaki, along with the Manufacturing Development Laboratory and the Industrial Electronics and System Development Laboratory. The *Materials and Electronic Devices Laboratory* is in Sagamihara to facilitate transfer to the Sagami Works of efforts in producing copper alloy products, including superconducting wires, all kinds of printed circuitry, a wide range of ferrite products, electronic equipment, and chemical products. The latter include many variations of composites. MELCO is proud that its composites experience began in the early 1950s.

Table 31 is a two-page list of recent papers by MELCO staff on various kinds of composites. MELCO provided us with reprints of the asterisked numbers. Table 32 describes current and anticipated applications, and Table 33 is a listing of current interests. MELCO is not making C-C at present because it does not have the requisite facilities.

We were shown several videos and slide shows, mostly emphasizing MELCO roles in defense (MELCO manufactures the Hawk and Patriot missiles under license from Raytheon) and in various Japanese space activities. MELCO uses a four-axis filament-winding machine to make space struts from C/epoxy and is trying to convert to MMC. The struts and joints of Communications Satellite No. 3 launched on NASDA's H-1 rocket

Table 31

RESEARCH ACTIVITIES ON COMPOSITES

MMC

1) Y. Kagawa, S. Utsunomiya, Y. Kogo ; Laser Cutting of CVD-SiC Fiber / A6061 Composite, J. Materials Science Letters, to be published

2) Y. Kogo, S. Yamashita, S. Utsunomiya ; Classification of Fracture Mechanism in SiC Whisker Reinforced Aluminum Alloy, Proc. Japan Inst. Met.,(1988.10), (in Japanese)

3) S. Utsunomiya, M. Okumura, S. Murakami, T. Morita; Fabrication Parameters in Laser Processing of MMC, Proc. Japan Inst. Met. (in Japanese), (1988.10)

*4) S. Utsunomiya, M. Okumura, S. Murakami, T. Morita, S. Hiramoto ; Fabrication of MMC Using Laser, 4th Japan-US Conf. Comp. Mat., to be published

*5) Y. Kagawa, S. Utsunomiya, Y. Kogo, M. Imaizumi; Fialure Behaviors of Two-Dimensionally Random-Oriented Short Carbon Fiber Reinforced Aluminum under a Stress Concentration, Proc. 6th ICCM, vol 2, (1987)

6) Y. Kogo, Y. Kagawa; Fracture Estimation and Strength of Whisker Reinforced Metals, Proc. Japan Inst. Met., (1987.10), (in Japanese)

7) S. Utsunomiya, Y. Kogo, Y. Kagawa; Cause of Residual Pores in Whisker Reinforced Metals made by Squeeze Casting, Proc. Japan Inst. Met., (1987.10), (in Japanese)

*8) S. Utsunomiya, Y. Kogo, Y. Kagawa ; Laser Induced Phenomena in Metal Matrix Composites, Proc. 3rd Japan-US Conf. Comp. Mat., (1986, 6), 589

9) Y. Kagawa, B. H. Choi (Tokyo Institute of Technology) ; Relation between Tensile Strength and Fracture Toughness in Brittle Fiber Reinforced Metals, Proc. 3rd Japan-US Conf. Comp. Mat., (1986, 6), 537

*10) S. Utsunomiya, Y. Kogo ; MMCs for High Temperature Use Antenna, Proc. Workshop on Structural Materials for HOPE, (1988.9), (in Japanese)

CMC

1) Y. Kogo, Y. Kagawa ; Fracture Behavior and Scatter of Strength in SiC Whisker Reinforced Glass Composite, Trans. Iron and Steel Inst. Japan, (in Japanese), to be published

2) Y. Kogo, Y. Kagawa; Mapping of Failure Process in Whisker Ceramics Composites Using Acoustic Emission Parameters, J. Mat. Sci. Letter, to be published

Table 31 (Cont'd)

3) Y. Kagawa ; Effects of Interfacial Shear Strength on the Fracture of Fiber Reinforced Ceramics, Trans. Iron & Steel Inst. Japan, Vol. 27, No. 1, (1987)

4) Y. Kagawa, M. Enoki(Univ. Tokyo), T. Kisi(Univ. Tokyo); Crack Propagation Studies in a Whisker-Glass Composite, Sintering '87, Tokyo(1987.11)

5) Y. Kagawa, Y. Kogo; Thermally Induced Phenomena in Whisker Reinforced Glass Composite, Sintering '87, Tokyo(1987.11)

6) Y. Kagawa, Y. Kogo; Acoustic Emissions from Whisker Reinforced Glass Composites, Sintering '87, Tokyo(1987.11)

C/C Composites

*1) Y. Kagawa, S. Utsunomiya, M. Imaizumi ; Deformation and Fracture Behavior of C-C Composite Having the Source of a Stress Concentration, Proc. 30th Japan Cogress Mat. Res., (1987), 195

3-D Fabric

*1) H. Hatta ; Elastic Moduli and Thermal Expansion Coefficient of Three Dimensional Fabric Composites, J. Japan Soc. Comp. Mat., Vol.14, No. 2, (1988), 73, (in Japanese)

*2) H. Hatta, K. Murayama ; Thermo-mechanical Properties of Three-dimensional Fabric Composites, Composite Structures 4, ed. by I.H. Marshall, Elsevier Applied Science, London (1987), 2.409

Hybrid FRP

*1) H. Hatta, S. Yamashita ; Fiber Orientation Control by Means of Magnetic Moment, J. Comp. Mat., Vol. 22, (1988)

Ceramic Redomes

1) T. Sone, K. Akagi, Y. Utumi ; Transparency of Hot-pressed Aluminum Oxynitrides, Proc. Cer. Soc. Japan Ann. Meet., (1988), 261, (in Japanese)

Table 32

COMPOSITE MATERIAL APPRICATIONS

APPRICATION	Type of Composites		Expected Characteristics
	Today	Future	
SPACE			
Spacecraft Components	CFRP	MMC	High TC
Antenna	ArFRP		Low CTE
Structure			Stability in
Solar Array Panel			Space Environment
Joint etc.			
Optical Mirror	CFRP	MMC/CMC	Dimentional Stability
HOPE/Spaceplane	–	MMC/CMC	Heat Resistance
DEFENSE			
Radar & FCS	CFRP	MMC	High TC
Antenna	GFRP		Low CTE
Radome			
Structure			
Missile	GFRP	CMC	Heat Resistance
Radome	Ceramics		

Table 33

Research & Development of Composites in MELCO

MMC

1) Joints for a satellite	High specific elasticity
2) Cases for electronic devices	Thermal expansion matching with ceramics
3) Antenna bodies for spaceplanes	High temperature use Dimensional stability
4) Laser processing of MMC	High productivity

CMC

1) Mirrors	Low weight Suvivability against hostile environment
2) Redomes	High temperature use High toughness
3) Antennas for spaceplanes	High temperature use

C/C

1) Basic study on properties

Concerning studies

1) Ceramic redomes	Transparency
2) Three dimensional fabric	Design flexibility Dimensional stability
3) Hybrid composites	Interlayer strength

were of epoxy reinforced with M40 fibers. The structure and beams for the solar arrays of Engineering Test Satellite No. 5 launched to GEO in 1987 were also FW CRP. The tubes and plates of the 25-foot frame of the Earth Resource Satellite No. 1 to be launched in 1992 will be CRP, but the joints are expected to be SiC_w/Al--the first Japanese application of MMCs in space (Table 34, Fig. 30). MELCO is also working on three approaches to a thermal protection system for the spaceplane HOPE, incorporating ceramic tiles over Al MMC, Ti honeycomb, or a Ni-based superalloy.

Plant Tour

The TAT was given an extended tour of the production lines for printed circuits. They are mostly automated and very impressive. One circuit board follows another so quickly that one expects it will overtake the other.

The TAT saw a space antenna of CRP being laid up, by hand. The autoclave was about 12 feet by 12 feet and was said to operate to 7 atmospheres and 300°C. The autoclave table was heated by circulating hot oil; this was said to help control the temperature in the autoclave to ± 2°C.

The TAT saw the SiC_w/Al space joints, only four of which have been made.

MELCO has a loom that can make up to 7D fabrics and shapes. MELCO weaves 3D tubing and is considering 3D C-fiber space-joint preforms.

MELCO claims no effort on thermoplastic matrices.

The TAT saw no work on CMCs. However, after repeated questions MELCO representatives reported some work using SiC and Si_3N_4 whiskers in borosilicate glass but said, "The climate is not yet right and the properties we have obtained are not attractive."

Table 34

METAL MATRIX COMPOSITE JOINTS FOR SPACE STRUCTURE

1. Abstract

NASDA (National Space Development Agency of Japan) has developed ERS-1 (Earth Resorce Satellite), which will be launched early in 1992. MITSUBISHI ELECTRIC CORPORATION takes charge of manufacturing the structure and innovative designs are applied to minimize its weight. MMC joints are used for the first time in Japan. Its structure is a square box which is composed by CFRP (carbon fiber reinforced plastics) frames and panels and MMC is applied as metallic joints where two CFRP pipes are crossed. The joints connect a set of CFRP pipes straightforward and the other pipe pierces the joints. MMC (metal matrix composite) gives metallic joints of space structures splendid elasticity and improves vibration properties of space structures. Up to this time the joints were made of aluminum alloy, whose elastic modulus is inferior to that of CFRP.

MMC, SiC whisker reinforced aluminum (SiC_W/7075) is fabricated by a squeeze casting technique and finished to the joint shapes using wire cutting and machining. SiC_W/7075 has superior elasticity and mechanical strength to monolithic aluminum alloy 7075. MITSUBISHI R&D efforts have concentrated on the reliability of mechanical properties of MMC and will make the promising materials actual as structural materials in space.

2. Materials

NOMINAL COMPOSITION OF EXTRA SUPER DURALUMIN 7075

Al	Zn	Mg	Cu	Cr
bal.	5.1-6.1	2.1-2.9	1.2-2.0	0.18-0.35

PROPERTIES OF SiCw / 7075

Structure	β-SiC
Length	10-100 μm
Diameter	0.05-0.5 μm
Density	3.19 g/cm³
Aspect Ratio	50-200

3. Fabrication Process

Whisker Preforming

MMC Fabrication

Squeeze Casting

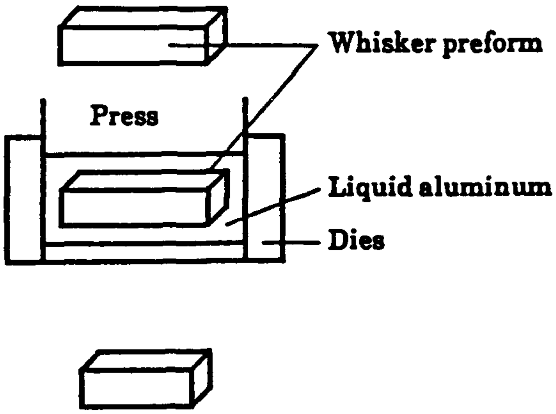
Machining

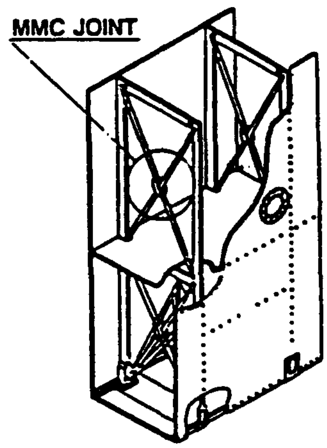
Wire Cutting
Machining

Assembling

Bonding

Products





THE STRUCTURE OF ERS-1

4. Mechanical Properties of MMC

PROPERTIES OF SiCw / 7075

Materials	Elastic modulus (GPa)	Tensile strength (MPa)	Fatigue strength at 10^7 (MPa)	Coeff. of thermal expansion (/K)	Density (g/cm^3)
SiCw/7075	106	570	>270	14×10^{-6}	2.88
Extra Super Duralmin	72	570	160	23×10^{-6}	2.80

Figure 30

H. TOA NENRYO KOGYO CO., LTD. (TONEN)

The TAT visited the Toa Nenryo Kogyo Co., Ltd. (Tonen) Corporate Research and Development Laboratory (CRDL) in Saitama on 9 February 1989 and Tonen's Kawasaki Refinery on 13 February 1989.

Tonen was established in 1939 as a producer of aviation gasoline and lubricants. In 1949 Tonen affiliated with Standard-Vacuum Oil Company, and today Esso Eastern, Inc. and Mobil Petroleum Co., Inc. each own 25 percent of Tonen (Fig. 31) and market Tonen's petroleum products via Esso Sekiyu K.K. and Mobil Sekiyu K.K. Tonen's major operations consist of the importation of crude oil and the manufacturing and sale of petroleum and petrochemical products from refineries at Kawasaki, Shimizu, and Wakayama.

Though Tonen is one of the largest (2400 employees) and most profitable petroleum companies in Japan, the Tonen Group emphasizes advanced technology and is developing new business in energy (ranging from new combustion systems to advanced solar cells), information science, life science, and advanced materials (principally pitch-based C fibers and Si_3N_4 fibers). The necessary research and development work is conducted at the Corporate R&D Laboratory on a 32 acre site in Saitama, about 20 miles northwest of central Tokyo. The R&D complex consists of several laboratory buildings plus four apartment buildings, two dormitory buildings, company dining rooms, lounges and studies, a gymnasium, several sports fields, and other facilities to provide a self-contained environment--a condition not uncommon to major Japanese facilities.

1. Pitch-Based C Fiber

At Tonen CRDL the TAT was given video and oral presentations on pitch-based carbon fibers. Table 35 illustrates the history of the pitch fiber development, based on the invention of low-viscosity liquid crystal pitch. As shown in Figs. 32 and 33, the liquid crystal pitch is obtained from FCC tar, a petroleum distillate residue, and spun into fibers that are subsequently carbonized at about 2700°F and graphitized at temperatures up to 5000°F. Table 36 gives the physical properties of three grades of fibers: high modulus (HM), ultrahigh modulus (UHM), and high (for pitch) tensile strength (HT). Figure 34 illustrates how Tonen sees its fibers in contrast to those of its major competitors. Tonen is especially proud that it can provide high-modulus fibers with higher strength and at lower

Subsidiaries

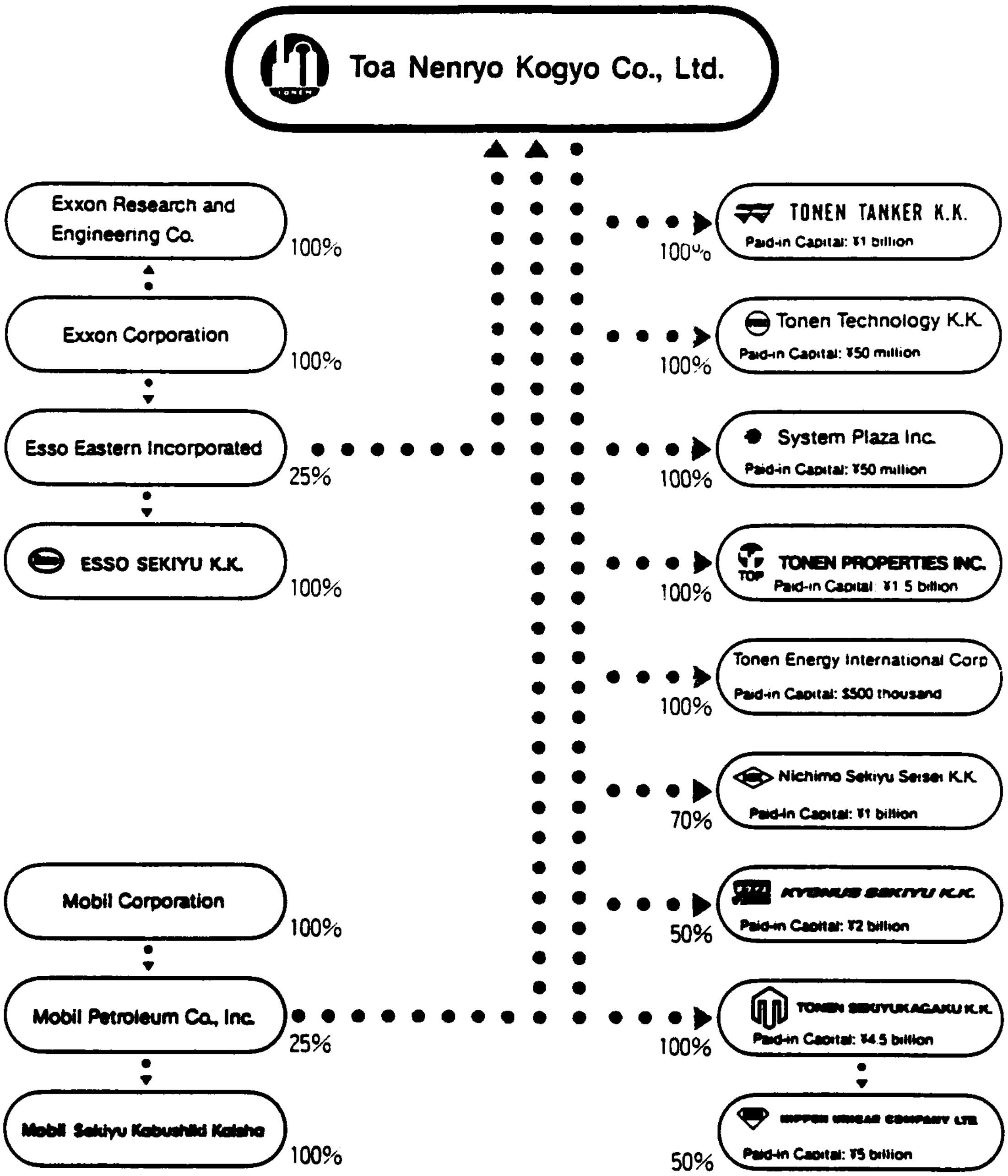


Figure 31

Table 35

DEVELOPMENT OF TONEN CARBON FIBER

1976～	EXPLORATORY RESEACH
1979	INVENTION OF LOW VISCOSITY LIQUID CRYSTALLINE PITCH
1980	BASIC PATENT APPLICATIONS IN MAJOR INDUSTRIALIZED COUNTRIES
1981～	BENCH SCALE DEVELOPMENT
1985	BASIC PATENTS GRANTED BY THE U.S.
1986	PRODUCT DEMONSTRATIONS BY PILOT PLANT
1987	PROCESS AND PRODUCT IMPROVEMENT
1988～	PRODUCT EVALUATION BY POTENTIAL CUSTOMERS

TONEN PITCH PROCESS

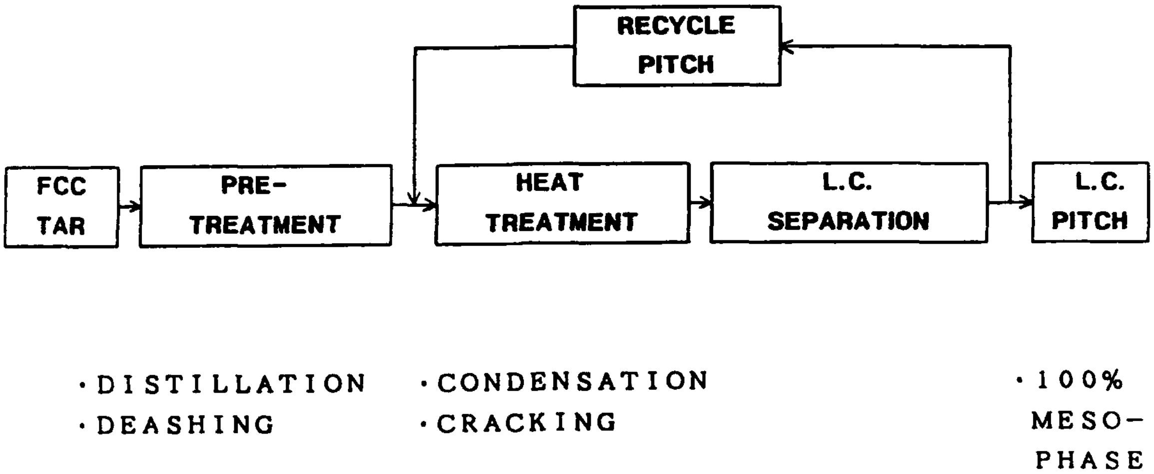


Figure 32

TONEN FIBER PROCESS

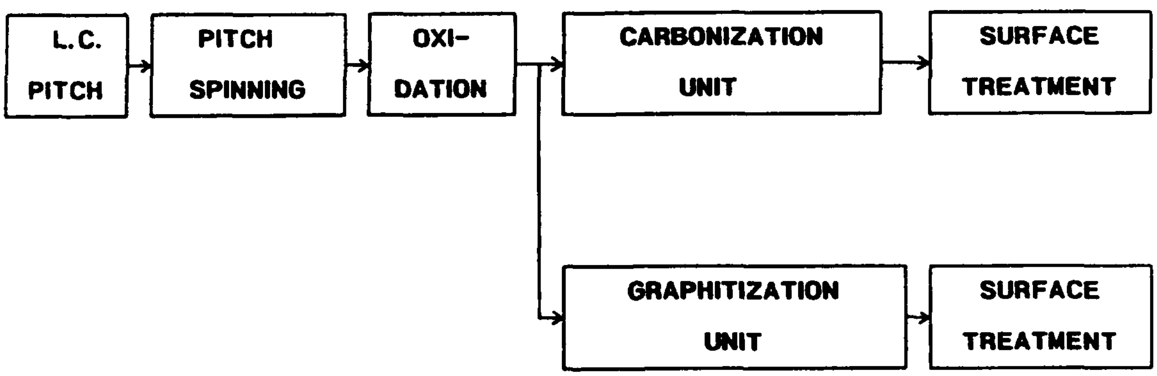


Figure 33

Table 36

PHYSICAL PROPERTIES OF TONEN CARBON FIBER

GRADE		UHM	HM	(HT) *
TENSILE STRENGTH	(KSI)	470	430	400
TENSILE MODULUS	(MSI)	100	70	36
ELONGATION	(%)	0.5	0.6	1.1
DENSITY	(g/cm^3)	2.16	2.14	2.05
DIAMETER OF FILAMENT	(μ)	10	10	10
YIELD as 6Kf	(m/g)	1.04	1.02	1.01

*Limited availability.

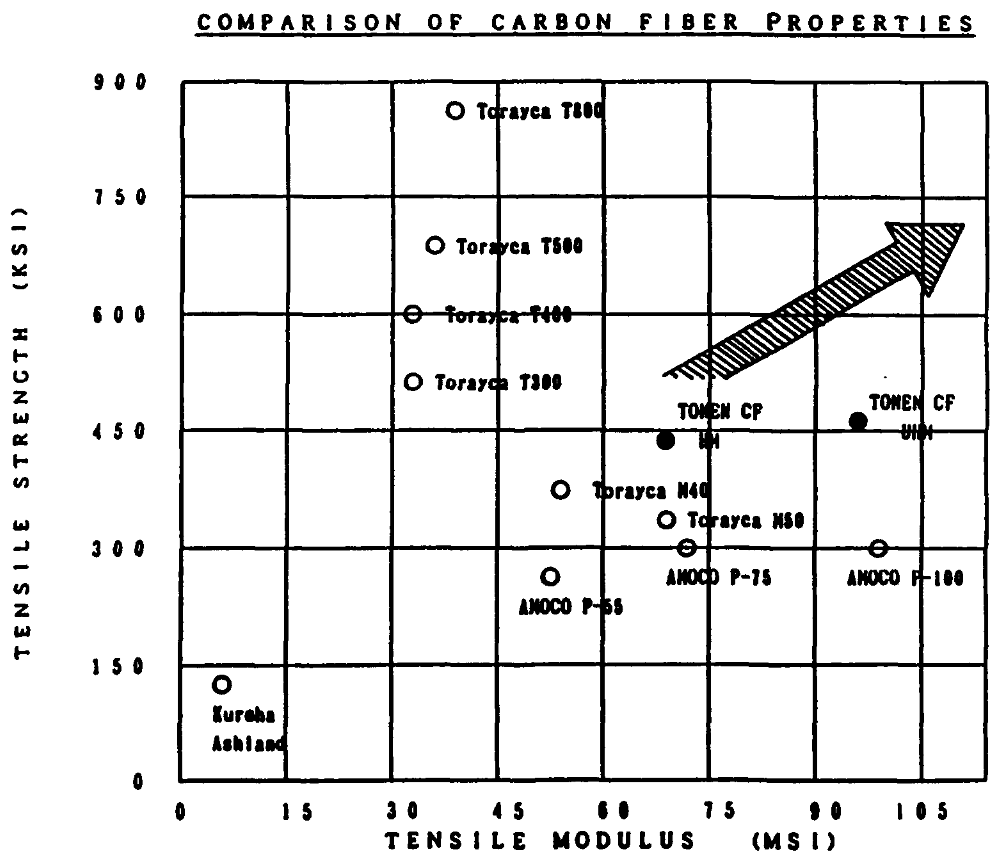


Figure 34

cost than the competition. Tonen would very much like to compete wtih PAN-based fibers in cost and strength. Tonen staff know that orientation and minimizing the ash content in the pitch are important, and they believe that spinning technology is critical. At present they are using electrolysis and oxygen-containing functions for fiber finishes and produce tows with 250 (for thin cloth), 3000, and 6000 ends. They are currently producing about 1 ton/month in a pilot plant, are making prepregs, pultruding beams, and pipes, and are doing some filament winding. Tonen is very eager for business arrangements in the United States and Europe; it sees the U.S. defense industry as a major market. Tonen expects to produce 5 to 20 tons/month in a prototype production facility in 1991-92 and hopes to be able to build a commercial plant (100 tons/month), maybe at a foreign site, in the mid-1990s.

At the Kawasaki Refinery on 13 February 1989, before a tour of the pitch-fiber facility, the TAT was shown an introductory video. Ukishima is an 1100 acre industrial complex on land reclaimed from Tokyo Bay; Tonen uses 330 acres for its Kawasaki Refinery and Tonen Sekiyu Kagaku K.K. (TSK), producer of petrochemicals and commodity resins and plastics. After the video, the TAT was informed that much of what it was to see was highly proprietary, even within Tonen, and that the team members were the first outsiders ever to see and hear what followed.

What followed was unparalleled in the experience of any TAT member. Whenever a production parameter was not evident visually, full process details (temperature, pressure, drawing rate, finish type, etc.) were volunteered or provided in answer to questions. The tour started at the liquid crystal pitch production stage and advanced through the spinning stage, the stranding and oxidation stages, and then precarbonization, carbonization, and graphitization. The pilot plant after the spinning stage was about 250 feet long.

Once again, high quality and low price were emphasized as immediate goals for the HM and UHM markets, which Tonen predicts may be 10 percent of the final C fiber market. This includes space, sporting goods, and special industrial uses. Eventually, by process control and automation, Tonen hopes to compete in the PAN-based C fiber market too. Tonen is doing prepreg and PMC development work to acquire experience and, possibly, sales.

2. Silicon Nitride Fibers

Tonen began research for silicon nitride fibers on a modest scale in 1981, using a polysilazane polymer precursor. The pace accelerated in 1986 with MITI funding; apparently MITI provides about 65 percent of a ¥2 billion budget to be spent over 5 years starting in 1986. About 20 people are working on the project. A bench-scale synthesis plant was installed in 1988; laboratory-scale spinning was done at 400 grams/hour with a 100 hole spinnerette, and pyrolysis was done in conventional electric furnaces. Bench-scale spinning and pyrolysis units were scheduled for 1989.

To make the Si_3N_4 fibers, dichlorosilane, pyridine, and ammonia are reacted and then polymerized in a batch process (Fig. 35). The resultant polysilazane has a simple chemical composition, and the purity, Si/N ratio, and molecular weight are easily controlled. Then green fibers are dry-spun (rather than melt-spun, to control crosslinking), given a pyrolitic NH_3 pretreatment, and pyrolyzed into the final Si_3N_4 fiber. At the time of the TAT visit, the post-spinning NH_3 and pyrolysis steps were done in batches, but continuous processing had been demonstrated on single filaments. The polymer synthesis and spinning were done in a clean room, and Tonen reported plans to conduct the entire production operation in clean room conditions. Tonen reports a yield of about 90 weight percent from polymer fiber to ceramic fiber. Figure 36 is a Tonen data sheet for its Si_3N_4 fiber. Additional data are shown in Figs. 37 and 38. Tonen says its fiber retains its surface and shape after 1 hour in air at 1200°C, but loses 40 percent of its strength (versus a 70 percent loss for Nicalon).

Tonen was beginning to explore the use of the polysilazane polymer as a precursor for oxidation-resistant coatings, Si_3N_4 monoliths, and C-fiber-reinforced Si_3N_4 matrix CMC.

Synthesis of Polysilazane

Toa Nenryo (1985)

1st Step, $SiH_2Cl_2 + 2Pyridine \longrightarrow SiH_2Cl_2 \cdot 2Pyridine\ Adduct$

2nd Step, $SiH_2Cl_2 \cdot 2Pyridine\ Adduct + 3NH_3 \longrightarrow$

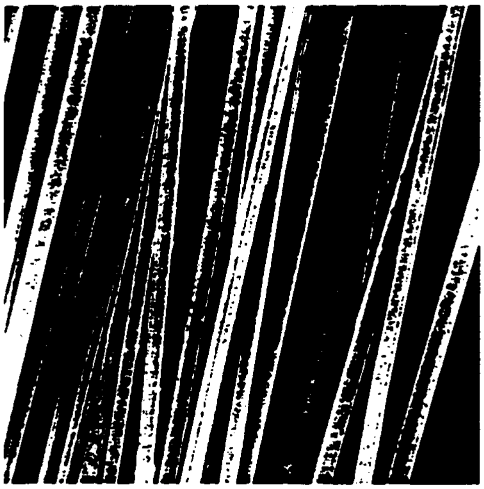
$$\{SiH_2NH\}_x + 2NH_4Cl + 2Pyridine$$

$$(SiH_2NH\}_x + NH_3 + \{SiH_2NH\}_Y \longrightarrow \begin{array}{l} -SiH-NH\{SiH_2NH\}_{x-1} \\ \quad | \\ \quad NH \\ \quad | \\ -SiH-NH\{SiH_2NH\}_{Y-1} \end{array} + 2H_2$$

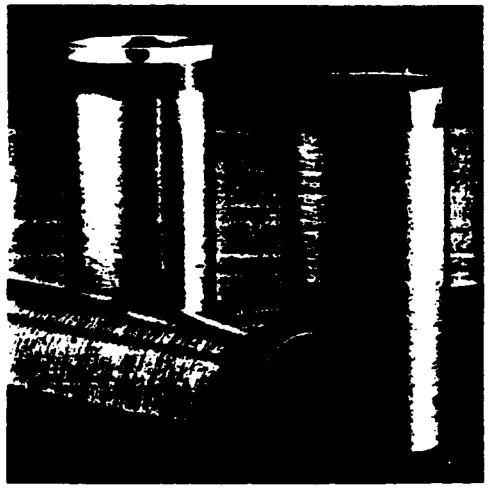
Figure 35

TONEN SILICON NITRIDE FIBER

Continuous Silicon Nitride Fiber, derived from our original preceramic polymer, is characterized by high purity, high strength and high heat resistance.



Photomicrograph of Silicon Nitride Fiber



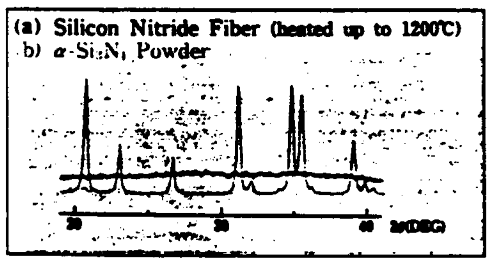
Silicon Nitride Fiber

Typical Properties of Silicon Nitride Fiber

Tensile strength (GPa)	2.5
Tensile modulus (GPa)	300
Filament diameter (μm)	10
Density (g/cm^3)	2.5
Maximum temperature for continuous use (°C)	1200
Crystal structure	amorphous

Composition of Silicon Nitride Fiber (wt%)

Si	N	C	O
59.2	39.1	0.5	1.1



X-ray diffraction patterns

TONEN
TOA NENRYO KOGYO K.K.
NEW BUSINESS DEVELOPMENT DEPT.
1-1 HITOTSUBASHI 1-CHOME, CHIYODA-KU, TOKYO, JAPAN
TEL. TOKYO 286-5195
FAX TOKYO 286-5120
TELEX 2227055 TONEN J

Figure 36

TG analysis of ceramic fibers

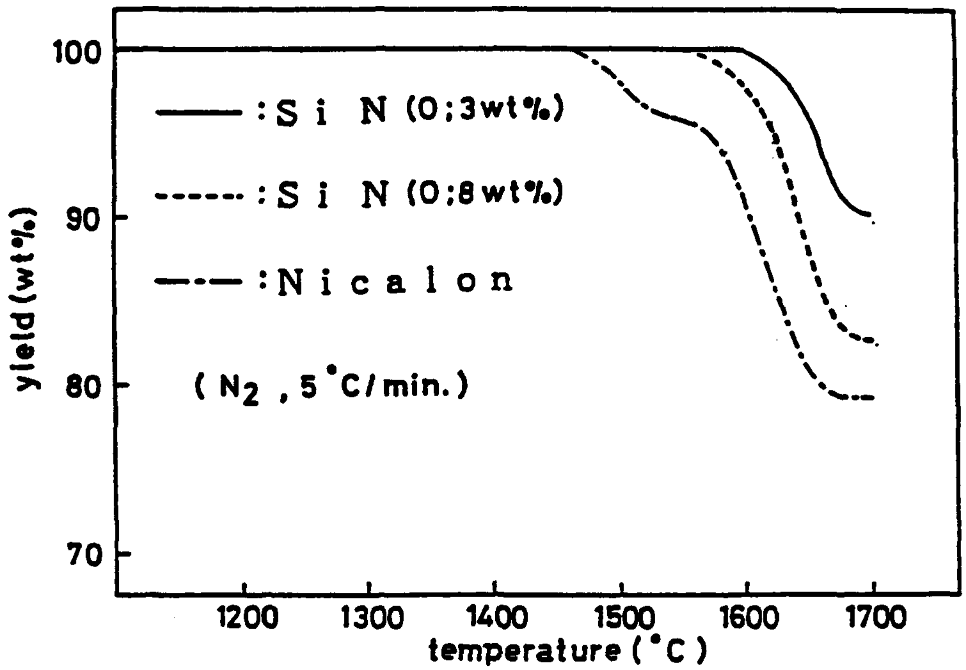


Figure 37

Oxidation Stability at Elevated Temperature

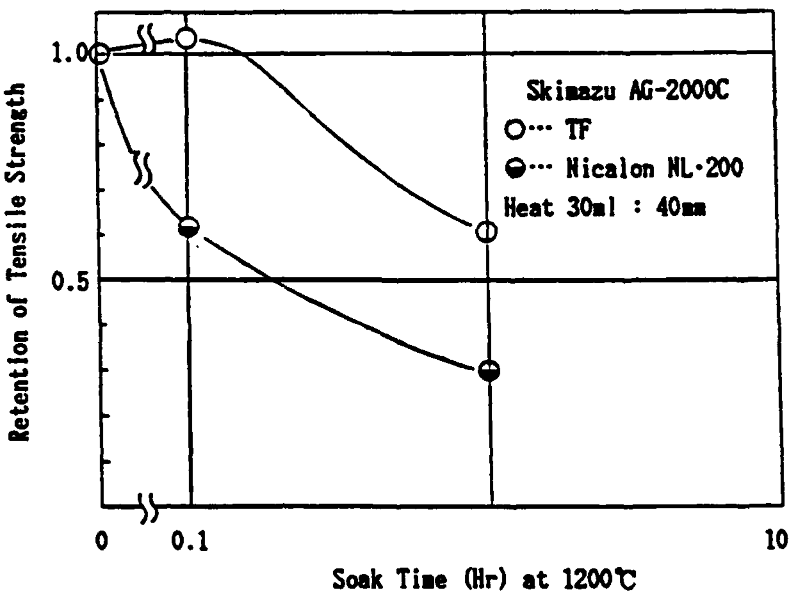


Figure 38

I. NIPPON CARBON CO., LTD. (NCK)

On 10 February 1989 the TAT visited the Yokohama plant of Nippon Carbon (NCK). NCK was established in 1915 to manufacture graphite electrodes, and to this day 70 percent of sales consists of artificial graphite electrodes for electric arc steel furnaces in 30 countries. As might be expected, the remainder of the product line is also carbon based, including corrosion-resistant components for the chemical and metallurgical industries, brushes for electric motors, and various bearings, seal rings, rotary vanes, etc. Carbolon carbon fibers are produced for heat-insulating materials and fillers for plastics and are resin-infiltrated for packings, gaskets, pump bearings, and valves. Nicafilm is a graphite film used for packings and gaskets in various industries. Overall, as the company chronology in Table 37 shows, NCK has been technology-oriented since its start, developing many new products over the years, beginning manufacture of carbon fibers in 1962, and getting a license to manufacture Nicalon in 1975.

1. Nicalon SiC Fibers

Table 38 recounts the R&D history of Nicalon fibers, from their invention by Professor S. Yajima at Tohoku University in 1975, through the initiation of R&D on MMCs and CMCs to the development of Nicalon fibers with high-volume and with low-volume resistivity. The process for making Nicalon fibers starts with the conversion of dimethyl dichlorosilane to a polycarbosilane polymer with a molecular weight of 1500 to 2000. Melt-spinning produces a very weak fiber which is made infusible by a 200°C cure in air. Subsequent heat treatment at 1300-1500°C in N_2 produces ceramic fibers 12-15 μ in diameter, consisting of 2 nanometer crystals of β SiC plus SiO_2 and C. The typical composition by weight is 58.3 percent Si, 30.4 percent C, 11.1 percent O, and 0.2 percent H. Calculated weight percents are: SiC 69.6 percent; SiO_2 20.8 percent; and C 9.6 percent. High-volume resistivity (HVR, also NL-400) fibers are said to be amorphous, and low-volume resistivity (LVR) fibers have 3 to 5 nanometer crystals. NL-300 fibers are 12 μ in diameter and about 10 percent stronger than the typical 14 to 15 μ fibers. Ten micron fibers with higher strengths have been made experimentally, as well as lower oxygen content fibers with higher modulus levels. Efforts are also under way to improve thermal stability.

Figures 39-42 are reproductions of Nippon Carbon data sheets that provide extensive information on the product forms, compositions, and mechanical and thermal

Table 37

INTRODUCTION OF NIPPON CARBON

PROFILE

Establishment:	28 December 1915
Total capital:	¥6,765,000,000
Number of employees:	825

CHRONOLOGY

1915 Nippon Carbon established in the city of Yokohama; commenced manufacture of natural graphite electrodes.
1926 Began to produce artificial graphite anodes for electrolysis.
1927 Manufactured the first artificial graphite electrode in Japan.
1932 Completed development of carbon brushes for electric motors.
1933 Construction of Yamanashi Plant for manufacture of graphite anodes.
1934 Construction of Toyama Plant for the manufacture of artificial graphite electrodes for arc furnace steelmaking.
1938 Construction of new Yokohama Plant for mass production of electric brushes and miscellaneous carbon products.
1949 Commenced production of impervious graphite "Resbon" for chemical applications.
1958 Began manufacture of nuclear graphite.
1962 Commenced manufacture of carbon fiber "Carbolon".
1966 Construction of Shiga Plant (Shin Nippon Carbon Company, Ltd.) as a subsidiary of Nippon Carbon for manufacture of artificial graphite electrodes.
1970 Commenced manufacture of high-strength and high-modulus carbon fiber "Carbolon-Z".
Shin Nippon Carbon Company Ltd. restarted as a joint venture with the Union Carbide Corporation of the U.S.A.
1974 Commenced manufacture of the flexible graphite seal material "Nicafilm".
1975 License obtained for manufacture of silicon carbide fiber "Nicalon".
1976 Nippon Carbon purchased all shares in Shin Nippon Carbon formerly held by Union Carbide Corporation and reorganized the joint venture as a wholly-owned subsidiary.
1980 Received grant from the Research Development Corporation of Japan for research and development in connection with silicon carbide fiber "Nicalon".
1981 Set up a joint venture with the Asahi Chemical Industry Company, Ltd., for the manufacture and sales of carbon fiber:
Asahi Nippon Carbon Fiber Company, Ltd.
1982 For market survey and marketing of Nicalon, we announced joint marketing in North America with Dow Corning Corporation, U.S.A.
1983 In order to expand the application of Nicalon, we also started a joint research program with Rolls Royce Limited, U.K. for the research and development of aluminum composites using Nicalon for the aero engine parts.
1985 Awarded the Deming Application Prize, Japan's most prestigious honor in the field of quality control.

Table 38

HISTORY OF RESEARCH & DEVELOPMENT OF SiC FIBER NICALON^{TM}

MAY, 1975	SiC FIBER(DISCONTINUOUS) WAS INVENTED BY PROF. S. YAJIMA AT TOHOKU UNIVERSITY.
JUN.	LICENSE OBTAINED FOR MANUFACTURING OF SiC FIBER.
APR. 1976	EXPERIMENTAL PLANT (DISCONTINUOUS FIBER).
DEC. 1978	SUCCESSFUL MANUFACTURING OF CONTINUOUS SiC FIBER.
	BENCH PLANT(CONTINUOUS FIBER, 25 KG/MONTH).
1979	STARTED R & D OF NICALON MMC.
MAY, 1980	SUPPORT FROM JAPAN RESEARCH DEVELOPMENT CORPORATION (JRDC) FOR R & D OF SiC FIBER.
FEB. 1981	PILOT PLANT(100 KG/MONTH).
APR.	R & D OF MMC WAS STARTED UNDER THE MANAGEMENT OF R & D INSTITUTE OF METALS & COMPOSITES FOR FUTURE INDUSTRIES, SPONSORED BY MITI.(1981 - MAR. 1989)

Table 38 (Cont'd)

OCT. 1982	MARKETING AGREEMENT FOR NICALON IN NORTH AMERICA WITH DOW CORNING CORPORATION, USA.
MAR. 1983	CONSTRUCTION OF NICALON 1 TON/MONTH PLANT.
DEC.	RECEIVED AUTHORIZATION FOR SUCCESS IN R & D OF SiC FIBER FROM JRDC.
1984	SUCCESSFUL DEVELOPMENT OF NICALON/Al COMPOSITE WIRE UNDER THE MANAGEMENT OF RDIMCFT, SPONSORED BY MITI.
	STARTED R & D OF NICALON/GLASS COMPOSITE.
1986	SUCCESSFUL DEVELOPMENT OF NICALON HVR (HIGH VOLUME RESISTIVITY) FIBER.
1988	SUCCESSFUL DEVELOPMENT OF NICALON LVR (LOW VOLUME RESISTIVITY) FIBER.
	R & D OF NICALON/CERAMICS COMPOSITE(NICALOCERAM™).

Applications of Nicalon®

- Use as a Reinforcer in Various Materials.

  Nicalon® can be used as a reinforcing fiber in metals, resins and ceramics for use in aeronautical, automobile, and nuclear furnace materials. It can also be used in industrial materials, audio parts and sports equipment.
- Use as a Heat-resistant Material.

  Due to Nicalon® having heat resistant and insulation properties in a high temperature atmosphere, it can be utilized in safety shields used in high temperature operations, in high temperature heat treatment furnaces, in filters for use in high temperatures, in packing for use in high temperatures and so on.

Finished products of Nicalon®

- Continuous fiber(multi-filament)
- Chopped
- Various Woven Products (cloth, braid, tape, rope)
- Various Composite materials (MMC, PMC, CMC)

Product code number of Nicalon® continuous fiber

NICALON® (Silicon Carbide Continuous Fiber) is available in the following types designed for various applications.

type:
- NL-200 series
- NL-300 series
- NL-400 series HVR

Depending on the application of Nicalon, a suitable sizing agent can be chosen.

Product Code Number	Type of Sizing Agent
NL-××0	none
NL-××1	use in PMC(EPOXY)
NL-××2	use in MMC. CMC
NL-××3	use in PMC(BMI)

Properties of Nicalon® yarn

		NL-200	NL-300	HVR NL-400
Diameter	(μm)	15	12	15
Numbers of Filaments	(fil./yarn)	500	500	500
Tex	(g/1000m)	210	140	210
Tensile Strength	(kg/mm^2)	280	300	250
Tensile Modulus	(10^3kg/mm^2)	20	20	18
Elongation	(%)	1.4	1.5	1.4
Density	(g/cm^3)	2.55	2.55	2.30
Specific Resistivity	(Ω-cm)	10^3	10^3	10^6
C.T.E.	(10^{-6}/°C)	3.1	3.1	3.1
Specific Heat	(J/g°C)	1.14	1.14	–
Heat Couductivity	(kcal/m·Hr·°C)	10	10	–
Dielectric Coustant		9	9	6.5
Specific Surface Area	(m^2/g)	0.13	–	–

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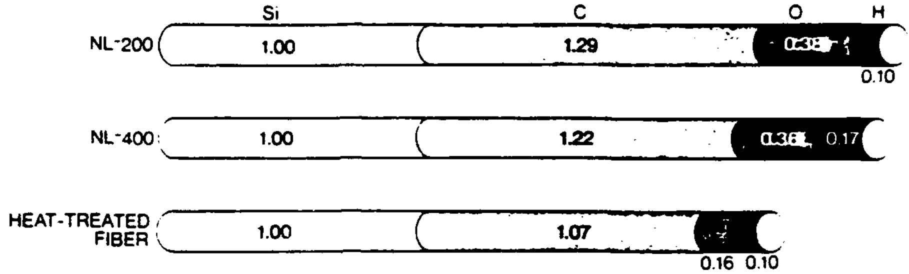
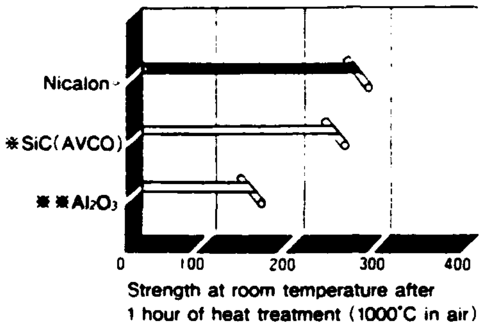
TEL: 03-552-6125 FAX: 03-555-8961 TELEX: 252-2665 NCKJ

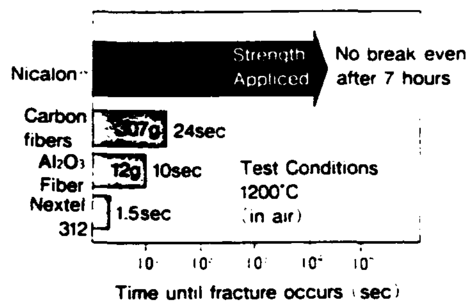
Figure 39

Composition (Atomic Ratio)



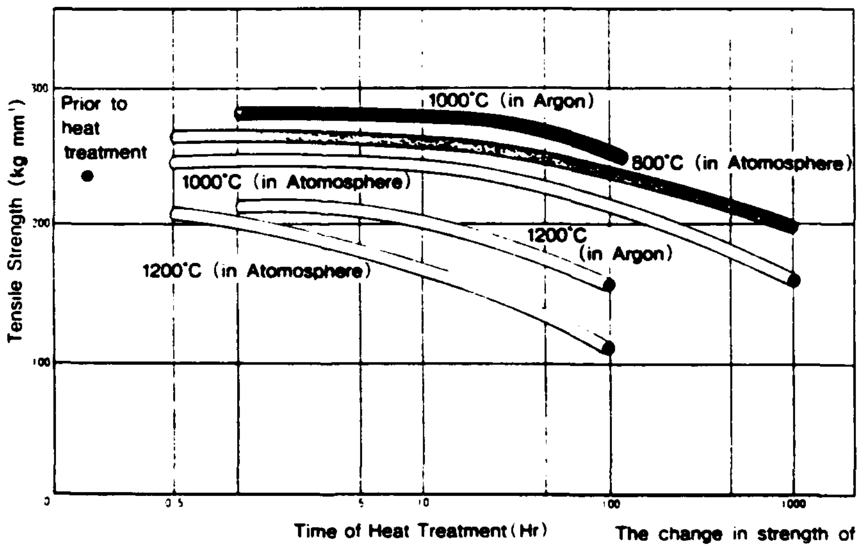
A comparison of heat resistance with other fibers





*AVCO Catalogue value
**Dupont Catalogue value

Nicalon
(Change in strength after heat treatment)



The change in strength of a Nicalon monofilament after heat treatment

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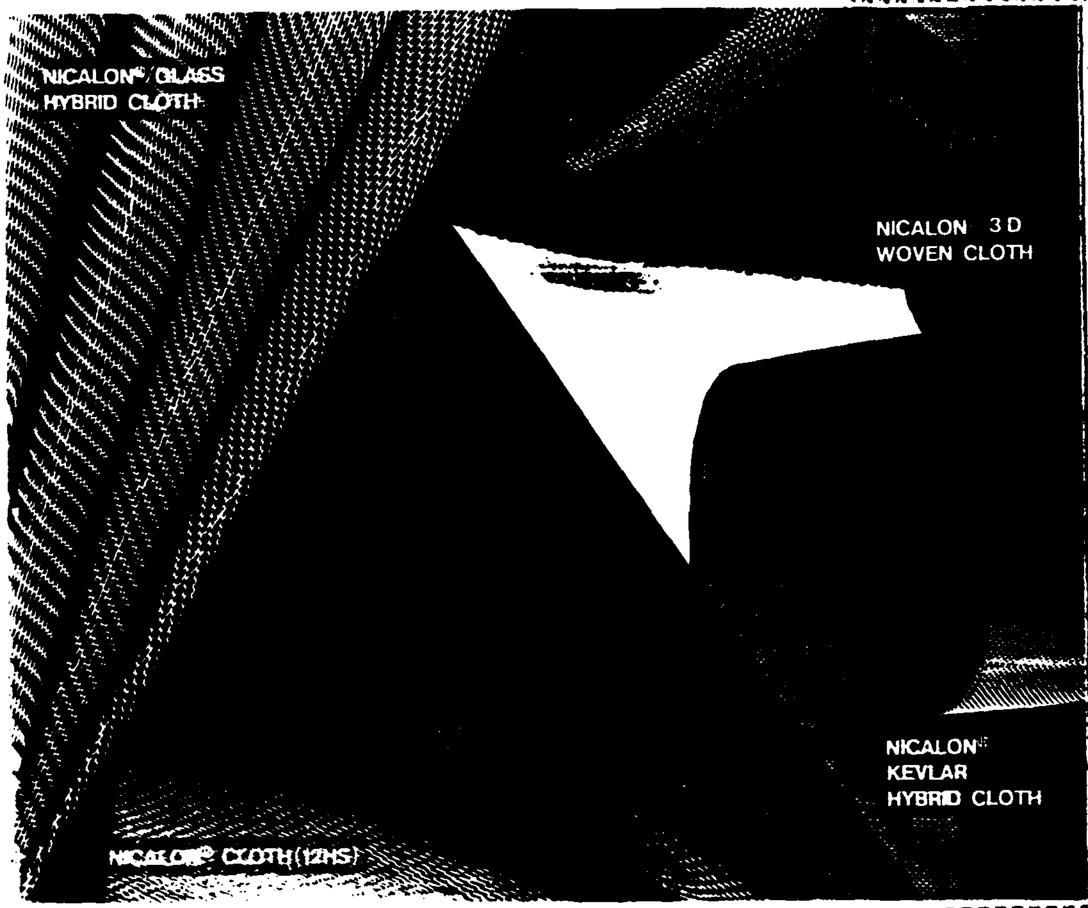
Figure 40

NICALON®

NICALON®(Silicon Carbide Continuous Fiber)

Woven Fabric

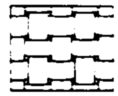
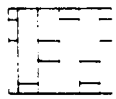
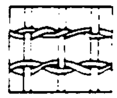
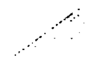
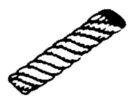


5 Major Features of Cloths

1. Due to excellent heat resistance properties and heat insulation properties, they can be used in a wide range of heat insulating materials for high temperature use.
2. Due to very good oxidation resistance, it is possible for steady and stable use over a long period of time.
3. Due to them being flexible and and easy to handle, it is possible to use them in various ways.
4. These cloths show extremely high stability, even with molten Aluminum and Copper.
5. They snow good wettability (compatibility) for resins, meaning that they can be applied in various composite materials.

Figure 41

Specifications of Nicalon® cloths

	Weave	Product Code Number	Specifications: Amount of yarns used	Specifications: Weight	Specifications: Meas-urements	Tensile (kg) strength at breaking point
	Plain	NL-201-PL NL-202-PL	warp : 16yarn inch weft : 16yarn inch	280g/m^2	w: 1000mm ℓ: 50m t: 0.28mm	warp : 130 weft : 150
	8 Harness Satin	NL-201-8S NL-202-8S	warp : 22yarn/inch weft : 22yarn/inch	380g/m^2	w: 1000mm ℓ: 50m t: 0.35mm	warp : 180 weft : 190
	12 Harness Satin	NL-201-2S NL-202-2S	warp : 26yarn/inch weft : 26yarn/inch	450g/m^2	w: 1000mm ℓ: 50m t: 0.45mm	warp : 210 weft : 240
	Reno	NL-201-LC NL-202-LC	warp : 5yarn/inch weft : 5yarn/inch	170g/m^2	w: 1000mm ℓ: 50m t: 0.45mm	
	*** Braid	NL-201-B2 NL-202-B2	Total: 48yarn Pitch: 6inch	35g/m	w: 20mm ℓ: 50m	
	*** Tape	NL-201-T1 NL-202-T1	warp : 35yarn/inch weft : 15yarn/inch	4.5g/m	w: 10mm ℓ: 50m t: 0.40mm	warp : 320
	*** Rope	NL-202-R5	100yarn	15g/m	d: 5mm	310
	Paper	NL-202-P		20g/m^2	w: 1000mm ℓ: 1000mm t: 0.25mm	
	Mat	NL-202-M		200g/m^2	w: 1000mm ℓ: 50m t: 2.3mm	

Note: *) With regards to length, we are prepared to make lengths to fit orders.
**) Evaluation made by JIS standard R-3420.
***) Braids, tapes and ropes are just examples of products. We are willing to produce products to suit orders.

● Due to Nicalon® being a flexible filament yarn, when a suitable amount of sizing is applied, it can be formed or woven into various cloths.
● Depending on its application, a sizing agent can be used.

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Figure 42

properties of Nicalon fibers. Table 39 provides more data on the HVR- and LVR-grade fibers, and Table 40 identifies the surface treatment options and intended uses. Table 41 provides data on NL-607, a carbon-coated Nicalon fiber intended to be used in CMCs.

It was reported that NCK invested about $22 million from 1975 to 1981 to develop Nicalon. Sales volume and price in 1988 were reported to be 6 tons and $600/kg.

2. Carbon/Graphite Fibers

NCK has been producing carbon and graphite fibers under the tradename Carbolon for almost three decades, beginning in 1962. Table 42 illustrates the various product forms. Apparently all of these materials are derived by heating organic fibers, but the precursor was not identified. For a period starting in 1981 NCK and Asahi Chemical Industry Co., Ltd. jointly produced a PAN-based C fiber called Carbolon Z, but sales were so slow that NCK withdrew. At present, NCK is producing 1 ton/month of pitch-based C fibers. Table 43 and Fig. 43 provide some data.

3. Metal Matrix Composites

NCK started its research into Nicalon-reinforced metals in 1979. MITI funding via the R&D Institute of Metals and Composites for Future Industries began in 1981 and continued to 1989. Figure 44 is a reprint of a summary paper provided to the TAT by NCK; it provides extensive information on processes, properties, and products. Figures 45 and 46 are reproductions of NCK data sheets that provide additional information on Nicalon/Al wire and composites.

4. Ceramic Matrix Composites

NCK initiated R&D of Nicalon-reinforced glass and glass ceramics in 1984. Figure 47 illustrates the process, and Table 44 and Figs. 48-50 provide some data. Figure 51 provides comparative data on stress intensity factors and flexural strength for numerous ceramics and CMCs.

In 1988 NCK began a study of a new CMC it calls Nicaloceram, in which Nicalon fiber reinforcements are combined with a polycarbosilane (similar to the Nicalon precursor) to make a SiC/SiC composite. Figure 52 illustrates the process, and some typical properties data are provided in Table 45. Figures 53 and 54 provide some elevated temperature data. Figure 55 is an NCK data sheet on both kinds of CMC.

Table 39

NICALON™ CERAMIC FIBER (Ceramic Grade, HVR Grade, and LVR Grade)

Type	Multifilament, textile-grade silicon carbide fiber
Color	Black
Physical Forms	Continuous, multifilament tow; chopped fiber; woven cloth
Fiber Denier	600, 900, 1200 and 1800
Surface Sizings	Various
Special Properties	High strength and modulus; high-temperature stability; tailorable electrical properties; weaveable
Primary Uses	Reinforcement in plastic, ceramic and metal matrix composites; high-temperature insulation and materials handling

TYPICAL PROPERTIES
These values are not intended for use in preparing specifications.

	(NL-200)	(NL-400)	
	Ceramic Grade	*HVR Grade*	*LVR Grade*
Fiber Denier	600, 900, 1200 and 1800	1800	1800
Density, g/cm^3	2.55	2.32	2.45 to 2.55
Tensile Strength, Ksi	430	425	430
Tensile Modulus, Msi	28	27	28
Strain to Failure, average percent	1.5	1.6	1.5
Volume Resistivity, ohm-cm	10^3	$>10^6$	0.5 to 5.0
Dielectric Constant	9.2	6.4	–
Thermal Conductivity, Kcal/mhr °C, along fiber axis at room temperature	10	–	–
Coefficient of Thermal Expansion, °C^{-1}, along fiber axis, 0 to 900 C (32 to 1652 F)	4.0×10^{-6}	–	–
Specific Heat, J/g °C	1.14	1.14	–
Thermal Conductivity, Kcal/mhr °C	10	–	–

Specification Writers: Please contact Dow Corning Corporation, Midland, Michigan, before writing specifications on these products.

Table 40

TABLE III: SURFACE TREATMENT OPTIONS

Designation	*Type*	*Intended Use*[1]
M-Sizing	Polyvinylacetate	CMC, MMC
PVA-Sizing	Polyvinyl Alcohol (Water Soluble)	CMC, MMC
P-Sizing	Modified Epoxy	PMC
DCC-1 Surface Treatment	Dow Corning Proprietary	PMC, Low-Temperature TS
DCC-2 Surface Treatment	Dow Corning Proprietary	PMC, High-Temperature TS and TP

[1]PMC = plastic matrix composite; CMC = ceramic matrix composite; MMC = metal matrix composite; TS = thermosets; TP = thermoplastics.

Table 41

CARBON COATED NICALON FIBER FOR CMC

* Strength of CMC increases
* Observed fiber pull out in the fracture surface of CMC

Table Typical properties of Carbon Coated NICALON Fiber for CMC (under development, sample supply available)

Product No.		NL-607
Diameter	(μm)	15
Numbers of filaments	(fil./yarn)	500
Tex	(g/1000m)	210
Tensile strength	(MPa)	3000
Tensile modulus	(GPa)	200
Elongation	(%)	1.5
Density	(kg/m^3)	2550
Specific resistivity	(ohm-cm)	0.5
Sizing agent		Polyvinyl alcohol
Sizing content	(wt%)	0.5
		weavable

Table 42

Carbolon® Products

Tow

Product No.	Thickness	Width	Length	Standard Weight	Standard Packing	Other
GF-8-T	–	–	–	–	1 kg packing	–
GF-20-T	–	–	–	–	1 kg packing	–

Twisted Yarn

Product No.	No. of Strands	Width	Length	Standard Weight	Standard Packing	Twist Direction
GF-8-Y	3	–	–	1.2g/m	1 kg bobbin wrapped	S/right Z/left
TSZ	2	–	–	0.8	100m paper tape wrapped	Z/left

Chop

Product No.	Thickness	Width	Length	Standard Weight	Standard Packing	Other
GF-8-C	–	–	3mm	–	10kg packing	(Note 1)
GF-20-C	–	–	3	–	10kg packing	(Note 1)

Felt

Product No.	Thickness	Width	Length	Standard Weight	Standard Packing	Other
GF-8-3F	3mm	750mm	3,000mm	530g/m²	roll	–
GF-8-5F	5	1,100	5,000	510	roll	–
GF-8-7F	7	1,100	4,000	810	roll	–
GF-8-10F	10	700	2,000	800	roll	–
GF-20-3F	3	700	3,000	400	roll	–
GF-20-5F	5	1,050	1,900	440	roll	–
GF-20-7F	7	1,050	1,350	730	roll	–
GF-20-10F	9	700	1,000	600	roll	–

Cloth

Product No.	Thickness	Width	Length	Standard Weight	Standard Packing	Other
GF-8-P7	0.45mm	800mm	50m	200g/m²	roll	plain weave
GF-8-S9	0.72	800	40	300	roll	satin weave
GF-8-P1	0.70	800	40	340	roll	plain weave
GF-20-P7	0.40	790	20	165	roll	plain weave
GF-20-S9	0.67	780	13	240	roll	satin weave
GF-20-P1	0.65	760	13	270	roll	plain weave

Paper

Product No.	Thickness	Width	Length	Standard Weight	Standard Packing	Other
SH-35	0.25mm	1,250mm	400m	35g/m²	roll	–
SH-35Z	0.31	1,000	500	33	roll	–
SH-45Z	0.35	1,000	100	45	roll	–

Formed Heat Insulation Materials

Product No.	Maximum Dimensions	Bulk Density	Standard Packing	Other
FGL (Cylinder)	external diameter 1,000φ x height 600mm	0.25g/c.c.	wooden crate	–
FGL (Board)	width 1,100 x length 1,500	0.25	wooden crate	(Notes 2,3)
FGM (Disk)	diameter 1,100φ	0.40	wooden crate	–

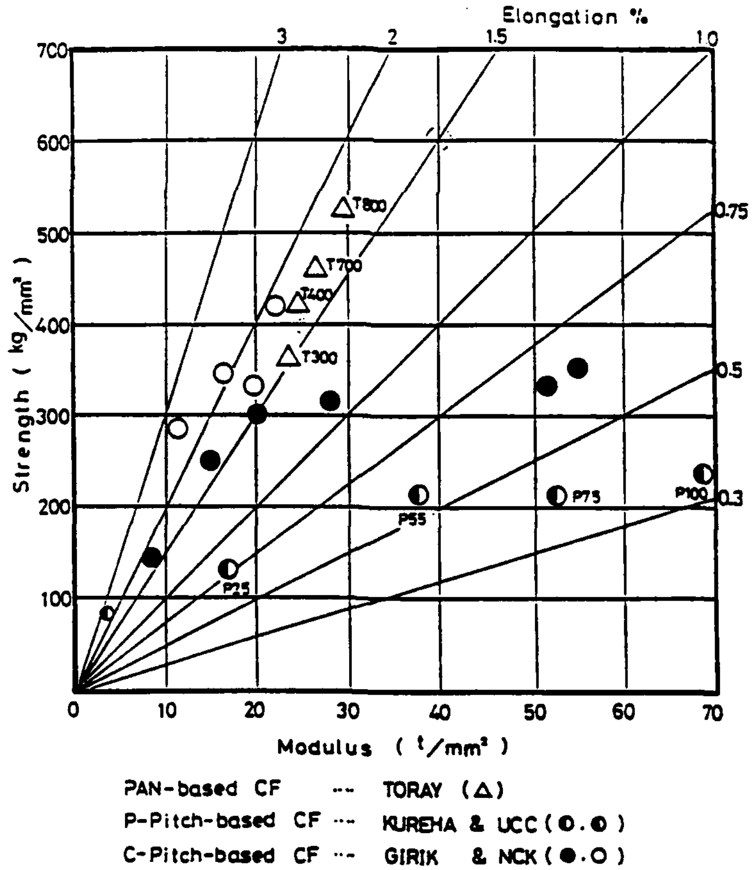
Notes:
1. Can be manufactured in lengths from 1mm to 70mm.
2. Please inquire concerning thickness and bulk density.
3. Longer lengths can be manufactured in separate lots.

Table 43

NIPPON CARBON Co. LTD.

PITCH-BASED HIGH PERFORMANCE CARBON FIBER

		PT-1	PM-1
Diameter	(μ)	10	9
Tensile Strength	(kg/mm^2)	250	350
Tensile Modulus	(t/mm^2)	15	60
Elongation	(%)	1.6	0.5
Specific Gravity		1.75	2.05
Strand Length	(m)	1,000	1,000
Filaments / Strand		2,000	2,000



Mechanical properties of carbon fiber

Figure 43

T21 FUTURE TRENDS IN SiC FIBER-REINFORCED METALS

T. ISHIKAWA

Nippon Carbon Co. Ltd.,
2-6-1 Hatchobori,Chuo-ku,Tokyo 104,Japan

Up until now, even though much has been expected of FRM(MMC), prospects of actual applications have been unclear. We consider the reason for this as being wholly due to the fact that practical fabrication methods for FRM composites have been absent. Under such circumstances, making the most of Japanese technology, in Japan emphasis has been placed on the development of FRM technology for fine, multifilament continuous fibers "such as carbon fibers and the SiC fiber NICALON" to be used mainly as reinforcements. This national project is planned to be carried out for the 8 years, 1981-1988, as part of the MITI's Project of Basic Technology for Future Industries. One feature of this has been the development of manufacturing technology of a "PREFORM WIRE", and the forming technology for composites applying this. The "PREFORM WIRE" here is already in a composite with aluminum, and is a half finished constituent used in forming.

At the present point in time, we have just managed to clear the project's targets of tensile strength 1.47 GPa at room temperature, and a heat resistant temperature of 450°C. We feel that this has solved over half of the problems regarding manufacturing methods facing FRM technology. In the future, it is hoped that this product can be docked with the concrete needs of actual users in a wide range of areas, bringing about an early age of practicability for FRM.

INTRODUCTION

In America and Europe, metal based composite materials are called MMC(Metal Matrix Composites), while in Japan they are called FRM (Fiber Reinforced Metals). This disparity in naming arises from different development ideology and is very interesting. American and European MMC are based on metallurgy and metal processing technology. That is to say, by applying already used metal processing technology a different kind of material is mixed into a metal to make a composite material. As a result, MMC made from reinforcements which do not match this method, for example fine multifilament continuous fibers like carbon fibers or the polycarbosilane(PCS) based SiC fiber "NICALON", (1-3) have never been made.

In Japan, as can be seen from the name FRM, emphasis in development has been placed on the reinforcing fiber itself. There are three reasons for this. Japan is a major producer of carbon fibers and is the only country in which SiC multifilament continuous fiber is produced. Also, the main reinforcing fibers of FRP (Fiber Reinforced Plastics - called PMC in America and Europe) are carbon fibers, or fine, multifilament continuous fibers. The third reason is that theoretically as well, composite materials made from continuous fibers should have superior properties compared to those made from chopped fibers, whiskers and powder strengtheners. This can also be understood from the fact that most CFRP utilize continuous fibers. (4)

The largest development project of FRM in Japan is taking place as a national project from 1981 to 1988. This came about because a FRM development project was included in the MITI's Project of Basic Technology for Future Industries. As is shown in Figure 1, this project consisted of two universities, four national research institutes and eight companies. In this project, along with carbon fibers the PCS based continuous fiber "NICALON" was also treated as an important reinforcing fiber. The target of this project

is shown in Table 1, and Figure 2. Briefly, the main aim was to produce a FRM while, although having similar lightweight, high strength and stiffness properties to FRP, could be utilized in a much wider temperature range.

This project's special structure came about from the largest problem regarding FRM, "How should we produce them?". We attempted to solve this problem by separating it into two parts, the compositing technology and the forming technology. It can be said that this was modeled after the relationship between prepreg and forming of FRP. In other words, development of a preform wire already made from the fiber and the aluminum matrix was established. Then, using this, a variety of forming methods were set up and carried out on it. As you can understand from this, the preform wire is a composite. (5)

It has been six years since this nation wide project commenced. At the present, a preform wire made from the PCS based SiC continuous fiber NICALON and aluminum has already cleared the target of this project. Progress in this development is shown in Figure 3. During this period, forming methods including hot pressing, roll forming and squeeze casting have been tested on the wire. Each produced an excellent FRM material, confirming the usefulness of the "preform" wire.

As from this year, this unique and superior preform wire has been made widely available, so new developments in a wide range of areas in Japan, America and Europe are anticipated. Below, follows a brief explanation of this new material "NICALON Preform Wire".

Fig. 1 JOINT FRM(MMC) BASIC INDUSTRIAL TECHNOLOGY R & D TEAM

AGENCY OF INDUSTRIAL SCIENCE AND TECHNOLOGY. MITI

PROGRAM STEERING COMMITTEE

PROGRAM COORDINATER

- MECHANICAL ENGINEERING LABORATORY
- GOVERNMENT INDUSTRIAL RESEARCH INSTITUTE. OSAKA
- INDUSTRIAL PRODUCTS RESEARCH INSTITUTE
- NATIONAL RESEARCH INSTITUTE FOR METALS.STA

R & D INSTITUTE OF METALS & COMPOSITES FOR FUTURE INDUSTRIES

- NIPPON CARBON CO.,LTD.
- TORAY INDUSTRIES. INC.
- ISHIKAWAJIMA-HARIMA HEAVY INDUSTRIES CO.,LTD.
- KAWASAKI HEAVY INDUSTRIES, LTD.
- FUJI HEAVY INDUSTRIES, LTD.
- MITSUBISHI HEAVY INDUSTRIES, LTD.
- MITSUBISHI ELECTRIC CORP.
- KOBE STEEL LTD.

- UNIVERSITY OF TOKYO
- KANAZAWA INSTITUTE OF TECHNOLOGY

Table 1. TARGET FOR FRM

(1) HEAT RESISTANT TEMPERATURE	450°C
(2) TENSILE STRENGTH*	1.47 GPa (150kgf/mm²)

* STRENGTH BY STANDARD SPECIMEN AND AT 90% RETENTION

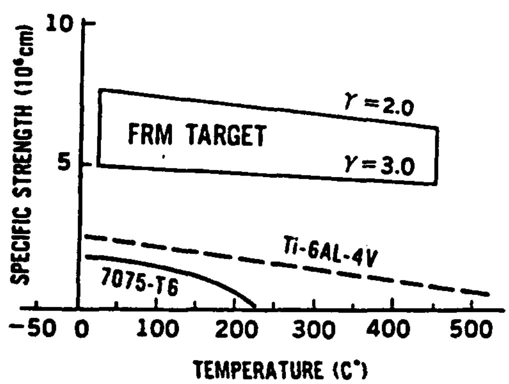


Fig. 2 TARGET AREA ON SPECIFIC TENSILE STRENGTH VERSUS TEMPERATURE DIAGRAM.

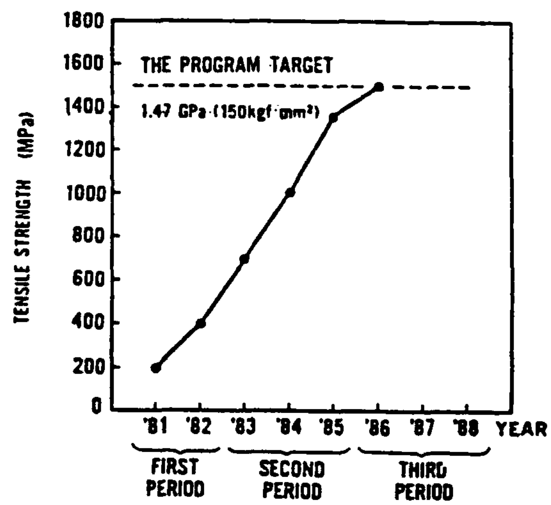


Fig. 3 PROGRESS IN THE DEVELOPMENT OF NICALON* AL "PREFORM WIRE"

The Reinforcing Fiber, "NICALON"

Basic technology for manufacturing NICALON was developed by the late Professor Yajima and his group, and industrial technology was developed by Nippon Carbon, making this development a truly Japanese effort. (6) Presently, this product is only being produced by Nippon Carbon, and a product like it cannot be found anywhere else in the world.

An outline of the manufacturing process is shown in Figure 4. The starting material, wide use silicone, is reacted, polymerized and converted to PCS (Polycarbosilane), an oligomer, with a molecular weight of 1,000-2,000. This is then spun and heat treated to form the SiC continuous fiber "NICALON". Some typical properties of "NICALON" are shown in Table 2. They include the following.

(1) This is a fine and flexible continuous fiber with an average fiber diameter of 12 micrometers and 15 micrometers. Because of this, a wide range of fiber processing can be undertaken, and composite materials with complicated forms can also be made.
(2) Suitable as a reinforcing fiber for lightweight materials as it has a low specific gravity.
(3) A higher tensile strength.
(4) A larger modulus of elasticity.
(5) Excellent oxidation resistance and stability at high temperature.
(6) There is no need for special surface treatment as it has good compatibility with metals.

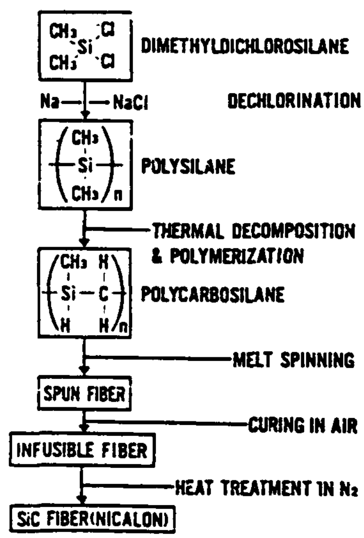


Fig. 4

THE PRODUCTION PROCESS OF POLYCARBOSILANE AND SiC FIBER NICALON®

Table 2 TYPICAL PROPERTIES OF SILICON CARBIDE CONTINUOUS FIBER NICALON®

PROPERTY	VALUE
FILAMENT DIAMETER	12, 15$\mu m \phi$
CROSS SECTION	ROUND
FILAMENTS / YARN	250, 500
TEX	70, 200g/1,000m
DENSITY	2.55 g / cm^3
TENSILE STRENGTH	2,500 ~ 3,000 MPa
TENSILE MODULUS	180 ~ 200 GPa
MAX USEABLE TEMPERATURE	1,250 C
COEFFICIENT OF THERMAL EXPANSION	3.1×10^{-6}/°C

Due to its above characteristics, NICALON has many applications. Besides the fiber itself being used as a gap filler in the space shuttle, it is also being utilized in a variety of heat resistant materials. As a FRP, it has been applied to micro wave absorbers, electro-magnetic welding pipes (general industrial use) and a wide range of sporting and leisure equipment. Also, recently, particularly in America and Europe, it has been used as the reinforcing fiber in FRC (Fiber Reinforced Ceramics, CMC) which use glass or SiC as their matrix. (7-10)

Due to reasons previously stated regarding utilization of fine continuous fibers in FRM, little of their research and development has been reported in America and Europe. In Japan, however, besides the national project mentioned above, studies by groups at Toyota Motor Corporation and other makers, some universities including Hiroshima University and the Toyama prefecture research group, have been undertaken. (11-13)

The NICALON / Aluminum Preform Wire

There is a tendency in all reinforcing fibers for a surface reaction to occur causing fiber degradation, when used with active molten aluminum. The fibers of NICALON are fine with an average diameter of 12 and 15 micrometers, about 1/10 of large diameter CVD method SiC fibers. Their cross-sectional area is about 1/100 of the latter. As a result,influence of surface reaction is much more sensitive than in the larger diameter fibers. This means that when making composites from fibers and a metal matrix, the following points should be paid close attention to :

(1) Deterioration of the fiber due to thermal and mechanical reasons.
(2) Wettability and reactivity of the fiber with the matrix metal.
(3) Uniform distribution of the fiber within the matrix metal.
(4) Defects and voids which occur within the composite.

These precautions are usually important when forming products. But the NICALON/aluminum preform wire is a composite wire which has overcome the above problems. In the production process, there is no need for intricate, unstable and expensive pre-treatments to be carried out. On top of this, infiltration of molten aluminum into the NICALON fiber bundles is sufficiently controllable, making this a very good process for mass production.
With an expansion in the demand of this product, a large decrease in the price will probably occur. It is thought that in the near future, a price below 100 dollars per pound will be possible.

If the main reason for a delay in practical applications of FRM is due to the difficulty and high cost of the manufacturing process, then it is anticipated that this preform wire has the ability to break down both these barriers. Some characteristics of the NICALON/ aluminum preform wire are introduced below. In Table 3 are shown some typical properties of Vf 45% and Vf 50% preform wires. The high strength Vf 50% wire clears the targets of the previously mentioned national project. The target being :

(1) Tensile strength at room temperature of 1.47 GPa.
(2) Tensile strength at 450°C of 1.32 GPa.

Table 4 shows a comparison of NICALON/aluminum preform wires and some commonly used structural materials. Superior specific strength and specific elasticity of the NICALON/ aluminum preform wire are quite clear. Generally speaking, composite materials are considered to be unreliable, but as the Weibull coefficient of the tensile strength has a high value of approximately 30, this wire can be said to be quite reliable.

Figure 5 shows the tensile fatigue characteristic of the preform wire. Compared with a non reinforced material, the fatigue characteristic figures are a lot higher. Whereas at 10 millions cycles, the figure for duralumin 2024-T3 is 300 MPa, the figure for the wire is twice that at 600 MPa. (14)

Table 3 PROPERTIES OF NICALON®-ALUMINUM COMPOSITE "PREFORM WIRE"

PROPERTY	VALUE	
CROSS SECTION	ROUND	ROUND
DIAMETER	0.5mm	0.3mm
TEX COUNT	550g/1,000m	170g/1,000m
TENSILE STRENGTH	1,200MPa	1,500MPa
TENSILE MODULUS E_1	130GPa	140GPa
TENSILE MODULUS E_0	80GPa	85GPa
FIBER VOLUME FRACTION	45Vol%	50Vol%
MINIMUM LOOP DIAMETER	30mm	20mm

(MATRIX : PURE ALUMINUM)

Table 4 CHARACTERISTICS OF NICALON® PREFORM WIRE AND TYPICAL STRUCTURAL MATERIALS

MATERIALS	DENSITY g/cm^3	TENSILE MODULUS GPa	TENSILE STRENGTH MPa	SPECIFIC STRENGTH 10^6cm	SPECIFIC MODULUS 10^8cm
AI SI 4340	8.0	200	1760	2.2	2.5
Al 7475	2.7	70	570	2.1	2.6
Ti-6Al-4V	4.4	110	1290	2.9	2.5
INCO 718	8.2	210	1430	1.8	2.5
17-7 PH.	7.8	200	1340	1.7	2.5
NICALON®/AL Vf50%	2.6	140	1500	5.7	5.4
PREFORM WIRE Vf45%	2.6	130	1200	4.6	5.0

4

Figure 44 (Cont'd)

Figure 6 shows the thermal fatigue characteristic of the NICALON preform wire. (15) A thermal cycle of -196°C to 400°C was repeated 200 times, but no changes in strength occurred. Results of elevated temperature resistance tests for exposure at 350°C and 450°C for 1000 hours are shown in Figure 7. Data from Figures 6 and 7 infer that this wire has very good thermal stability.

Also, in the following environment resistance tests, no change in strength was detected, showing good stability.

(1) Test at high temperature and moist :e (at 85°C - 90RH - 6Hrs)
(2) Boiling test (in boiling water - 100Hrs)
(3) Corrosion test in aqueous NaCl (in 5wt% NaCl Aq. - 20days)

FRM Using the NICALON Preform Wire

The properties of a hot-pressed (540°C, 35MPa, 20min.) FRM made from the preform wire are shown in Table 5. This wire is highly suitable when making composites with excellent strength and elasticity.

Using this wire, Mitsubishi Heavy Industries has succeeded in forming a plate(t2 x 300 x 300mm) by the quick hot press method, and Fuji Heavy Industries a sheet(t2 x 150 x 500mm) by the hot roll method. Both products display high properties, maintaining about 90-100 % of the tensile strength of the original wire. (16) Their samples can be seen in Figure 8. Both companies are continuing in their efforts to develop processes to make hybrid materials, long-sheet materials and so forth.

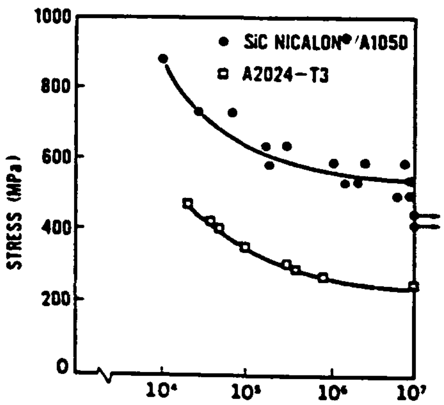


Fig. 5 TENSILE FATIGUE CHARACTERISTIC OF NICALON®/AL "PREFORM WIRE"

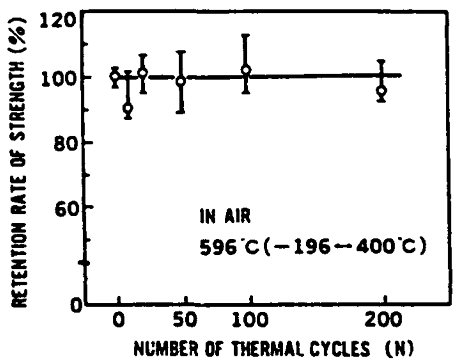


Fig. 6 THERMAL FATIGUE CHARACTERISTIC OF SiC(NICALON) Al "PREFORM WIRE"

Table 5 PROPERTIES OF SiC FIBER(NICALON®) /ALUMINUM COMPOSITE

(USING "PREFORM WIRE" BY HOT PRESSING)

PROPERTY	VALUE
FIBER VOLUME FRACTION	50 %
DENSITY	2.6 g/cm^3
TENSILE STRENGTH 0° / 90°	1350 / 70 MPa
FLEXURAL STRENGTH 0° / 90°	1450 / 120 MPa
TENSILE MODULUS 0°	E1 135 / E2 85 GPa
FLEXURAL MODULUS 0°	E1 125 / E2 80 GPa
POISSON RATIO	0.32
COEFFICIENT OF THERMAL EXPANSION 0° / 90°	8.8 / 25 $\times 10^{-6}/°C$

(UNI DIRECTIONAL)

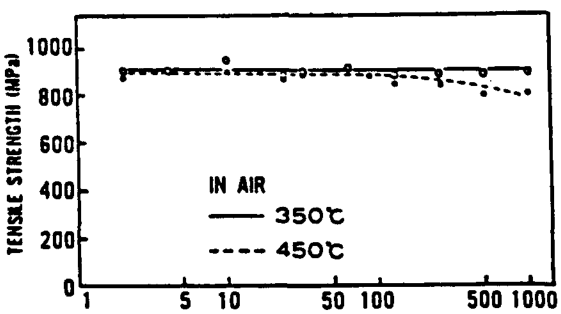


Fig. 7 EFFECT OF ELEVATED TEMPERATURE EXPOSURE OF SiC(NICALON) AL "PREFORM WIRE"

5

Figure 44 (Cont'd)

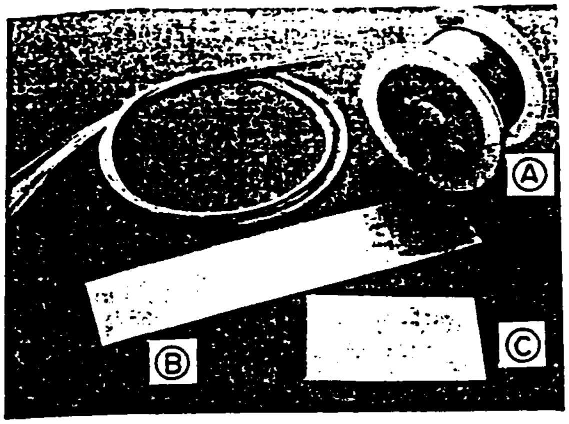


Fig.8 NICALON® "PREFORM WIRE" AND ITS FORMED MATERIALS

(Ⓐ PREFORM WIRES.
Ⓑ SHEET BY FUJI HEAVY INDUSTRIES
Ⓒ PLATE BY MITSUBISHI HEAVY INDUSTRIES)

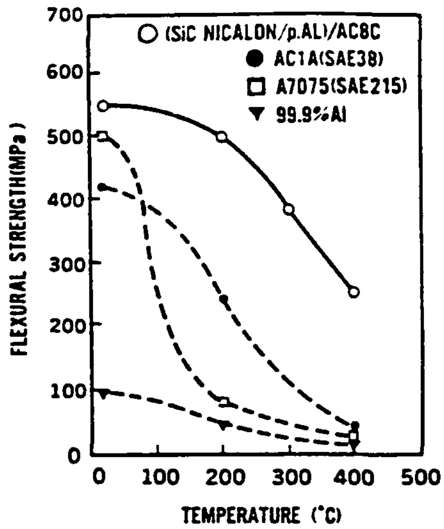


Fig. 9 FLEXURAL STRENGTH OF NICALON® "PREFORM WIRE" COMPO-COMPOSITE IN ELEVATED TEMPERATURE (UNIDIRECTIONAL,Vf 15%)

Also, a lot of energy is being put into the development of another type of FRM, where the preform wire is only inserted in the necessary position, making partially reinforced composites. First the preform wire is made into the shape of the place to be inserted. Next, this piece to be inserted is cast and the apertures between the wires are impregnated with molten aluminum. We call this a "Compo-Composite". Properties of a compo-composite made by a type of high pressure casting method are shown in Figures 9,10 and 11. The wire is arranged uniformly and unidirectionally to make a shape of a cylinder, and is then cast in an Al alloy. In the case shown, the wire used had a tensile strength of around 1,000 MPa, and Vf35%.

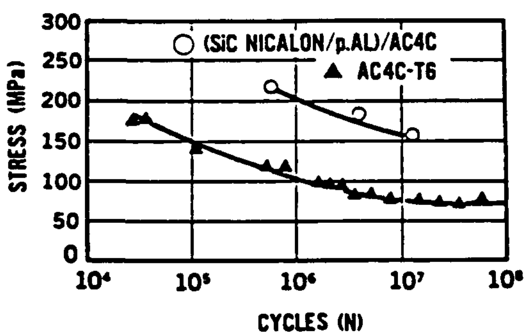


Fig. 10 ROLLING FLEXURAL FATIGUE CHARACTERISTIC OF NICALON "PREFORM WIRE" COMPO-COMPOSITE (UNIDIRECTIONAL,Vf 15%)

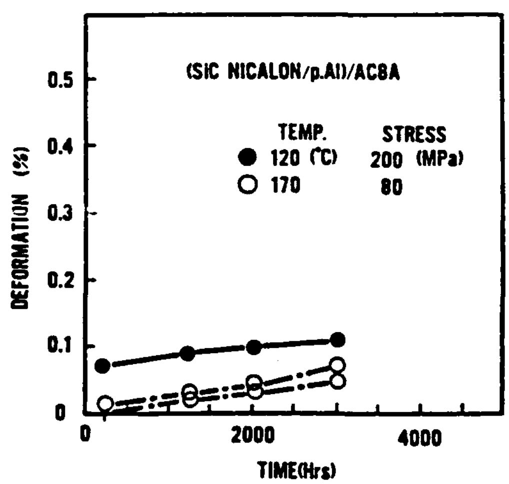


Fig.11 RESULT OF CREEP TESTS OF NICALON "PREFORM WIRE" COMPO-COMPOSITE (Vf 15%)

6

Figure 44 (Cont'd)

Due to fabrication of a compo-composite Vf decreased to 15%. However, as can be understood from Figures 9-11, it has excellent high temperature strength, fatigue characteristics and creep properties, showing sufficient fiber reinforcing effects.

Some examples of compo-composites are shown in the photographs. Figure 12 shows reinforcement of the sliding parts of a cylinder block used in an automobile air compressor. Figure 13 is the casing of an automobile differential gear. These are compo-composites made by an improved die-casting method. Figure 14 shows a connecting rod made by a type of high pressure casting method. Besides these, the shaft of a high speed rotator and others have also been made. The use of compo-composite materials, mainly in general industrial equipment, their forming method and performance appraisal are being considered at the moment.

Some characteristics of compo-composite materials are as follows :

(1) As the preform wire is used partially, only a small amount is necessary making them a lot cheaper.
(2) High productivity methods, like the die-casting method, can be utilized enabling a high production of compo-composites.
(3) As well as high strength and high elasticity, they display a range of functional characteristics including heat resistant stability, dimensional stability and anti-abrasiveness.
(4) Preform wires have a certain degree of flexibility, enabling a comparatively free design in their shape or form.

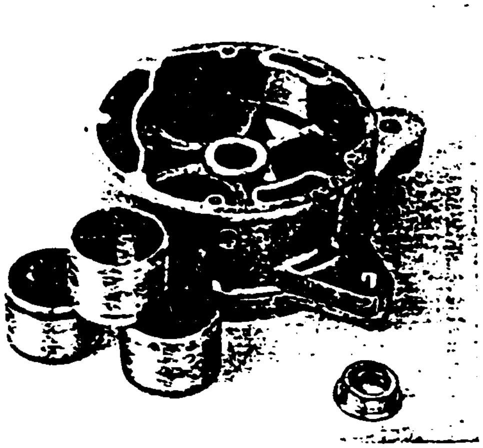


Fig 12 SAMPLE OF NICALON® "PREFORM WIRE" COMPO-COMPOSITE
(CYLINDER BLOCK FOR CAR AIR CONDITIONER)
(BY DIE CASTING METHOD)

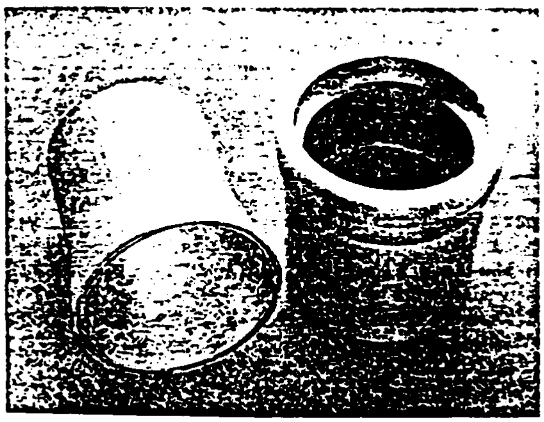


Fig 13 SAMPLE OF NICALON® "PREFORM WIRE" COMPO-COMPOSITE
(CASING OF DIFFERENTIAL GEAR FOR CAR)
(BY DIE CASTING METHOD)

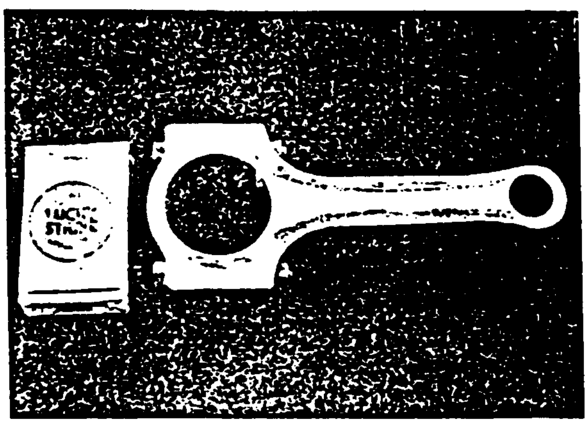


Fig 14 SAMPLE OF NICALON® "PREFORM WIRE" COMPO-COMPOSITE
(CONNECTING ROD FOR CAR)
(BY DIE CASTING METHOD)

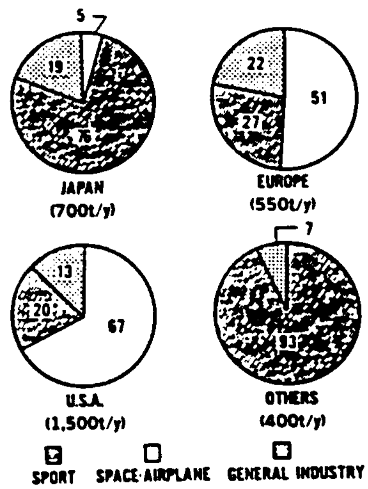


Fig.15 DEMANDS OF CARBON FIBER
(1985, 3150ton)

7

Figure 44 (Cont'd)

Conclusion

In this report we have introduced the fruit of new Japanese technology, the SiC continuous fiber NICALON, and its aluminum composite "NICALON preform wire". The reason for doing so, is not just because this is a new product, but because we think that as this NICALON preform wire does not face many of the problems faced by carbon fiber/aluminum composites - for example, electric corrosion, high temperature oxidation corrosion and the need for expensive pre-treatments like those used for CVD's - it is the most promising constituent for FRM(MMC) to be found so far. As this is a new material, at the present time, its applications and probable demand have not been fully grasped, but nobody can doubt the fact that a lot is expected of it in the future. Next, we take a look at some probable future developments of this product.

The demand distribution of carbon fiber FRP which are the spearhead of Advanced Composite Materials, is one good reference when considering the future of the preform wire. In Figure 15, demand areas for CFRP in Japan, America and Europe are shown. In the aerospace industry which is large in America and Europe, about 60% of CFRP is utilized in just this one area. On the other hand, in Japan where this area is minimal, 95% is used in the sports and general industry area. The same can probably be said regarding FRM. As a result, a lot of expectation is being placed on application developments of the preform wire in the aerospace industry in America and Europe. Table 6 shows some of these anticipated uses. (17) In Japan, a wide range of applications are anticipated in general industries, for example the automobile industry, as well as the energy and sports areas. (18-19)

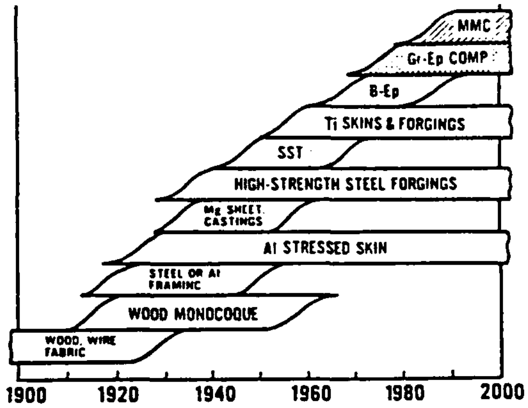


Fig.16 TRANSITION IN AIRCRAFT MATERIALS

Table 6 POTENTIAL COMMERCIAL APPLICATIONS OF SiC FRM FOR TRANSPORTATION

APPLICATION \ DESIRED PROPERTIES	LIGHT WEIGHT	STIFFNESS	STRENGTH	FLEXIBILITY	IMPACT RESISTANCE	HEAT RESISTANCE	RESISTANCE	EROSION RESISTANCE
AEROSPACE								
SPACE STRUCTURES	O	O					O	
ANTENNAE	O	O					O	
AIRPLANES								
PYLONS	O	O				O		
STRUTS	O	O	O					
FAIRINGS	O	O						
ACCESS DOORS	O	O	O					
WING BOX	O	O	O					
FRAMES	O	O	O					
STIFFENERS	O	O	O					
FLOOR BEAMS	O	O	O					
FAN AND COMPRESSOR BLADES		O	O		O	O		
TURBINE BLADES		O	O		O	O		O
HELICOPTERS								
TRANSMISSION CASES	O	O	O					
TRUSS STRUCTURES	O	O	O					
SWASH PLATES	O	O	O					
PUSH RODS	O	O	O					
TRAILING EDGE OF TAIL ROTOR BLADES	O	O	O					
AUTOMOTIVE								
ENGINE BLOCKS	O	O	O			O		
PUSH RODS	O	O	O			O		
FRAMES	O	O	O					
SPRINGS	O		O	O				
PISTON RODS	O	O	O					
BATTERY PLATES		O						

(ASTM STP704)

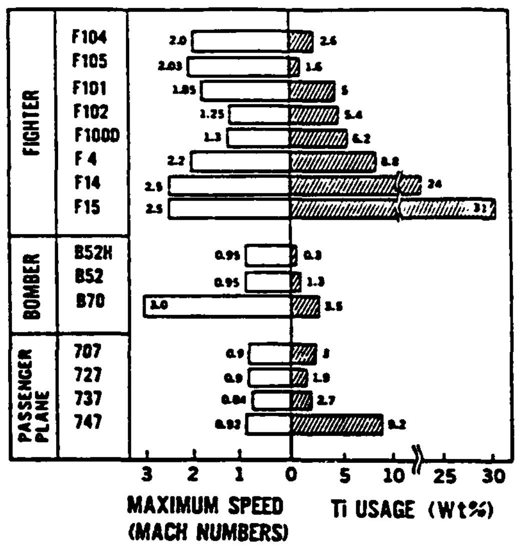


Fig.17 WEIGHT OF Ti USED IN PLANES

Figure 44 (Cont'd)

Figure 16 shows the transition which has occurred in aircraft materials. (20) It should be noted that second to carbon fiber/epoxy resin FRP, the newest material from which much is expected are MMC(FRM). Due to advancements in the performances of aircraft, especially in fighters, maximum speed (Mach number) has increased somewhat. Along with this, the amount of lightweight and heat resisting titanium used has also increased. This is shown in Figure 17, and is also the reason why we hope that NICALON/aluminum FRM can become a substitute for titanium. Moreover, materials for use in jet engines require high heat resistant properties and, as is shown in Figure 18, much is expected of MMC(FRM) and CMC(FRC). (21)

Another point of interest is the following. In "Chemical Abstracts" (1982-1986 ; Vol. 96-105) the number of extracts relating to silicon carbide fibers (excluding CVD's fibers and whiskers) is increasing year by year. Figure 19 shows that this number has undergone a sharp rise recently. Also, the fact that over the past year "Polycarbosilane", the spinning material for NICALON, has been independently listed in the index shows that interest in this area is on the increase. In the latter half of 1986, there were 47 such cases in "Chemical Abstracts", 30% (14 cases) being fiber related, while 60% (28 cases) were related to applications of FRM and FRC.

We feel that this is proof that expectations placed on the SiC fine continuous fiber NICALON's FRM are taking off and are on their way to becoming reality. We hope that composite technicians throughout the world will make efficient use of this new wire, and establish forming and appraisal technologies for FRM at the earliest possible opportunity. FRM development up until now has focused on original technology and not on customers' needs, but we think that it is now the time for users to aggressively indicate some concrete targets of what they actually want. By doing so, we feel that the coming of an age when FRM will be utilized practically and on full-scale may be greeted a little earlier than expected.

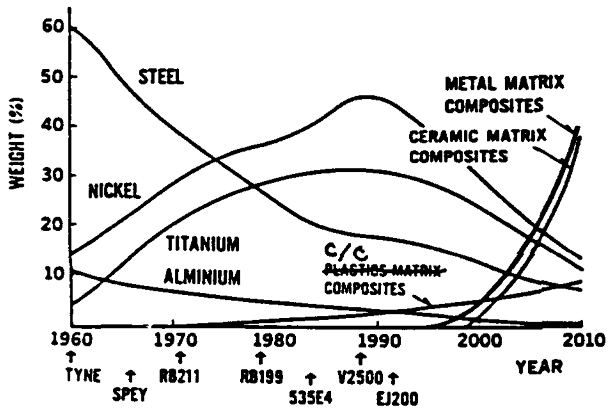


Fig.18 TRENDS IN JET ENGINE MATERIAL USAGE

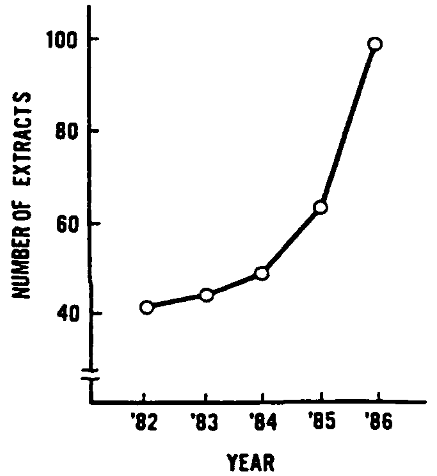


Fig.19 NUMBER OF EXTRACTS IN "CHEMICAL ABSTRACTS" ON SiC FIBER AND ITS APPLICATIONS (EXCLUDING CVD'S SiC FIBERS AND SiC WHISKERS)

Supplementary Notes

This work was partially performed under management of the Research and Development Institute of Metals and Composites for Future Industries sponsored by the Agency of Industrial Science and Technology, MITI.

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Figure 44 (Cont'd)

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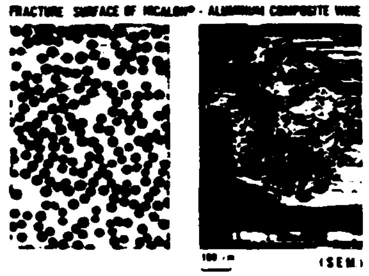
10

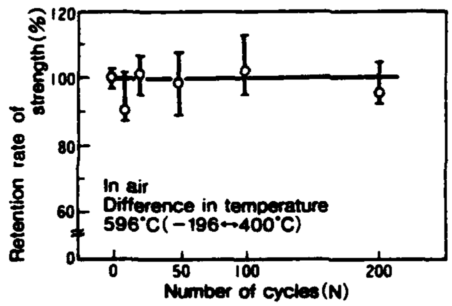
Figure 44 (Cont'd)

Typical properties of Nicalon® /Al composite wire

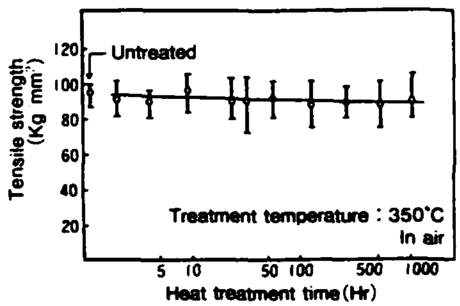
Item		
Cross-section		round
Diameter	(mm)	$\phi 0.5$
Count(TEX)	(g/1000m)	600
Tensile strength	(kg/mm²)	100
Tensile Modulus	(10^3 kg mm²)	13
Vf	(vol%)	40
Minimum diameter achieved	(mm)	$\phi 30$



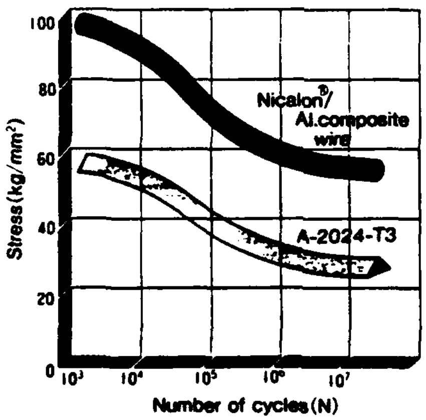
Various properties of Nicalon®/Al composite wire



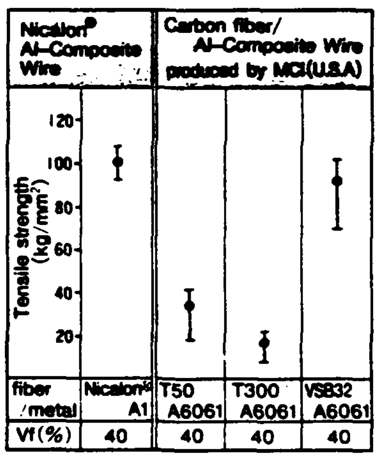
Repetitive heat cycle properties



High-temperature resistance properties



Tensile fatigue properties



Comparison of the tensile strengths of various Al. Composite wires.

⊙NIPPON CARBON CO., LTD. Head Office: 6-1, Hatchobori 2-chome, Chuo-ku, Tokyo, Japan TEL: 03-552-6125 FAX: 03-555-8961 TELEX: 252-2665 NCKJ

Figure 45

Properties of Nicalon®/Al(A–1050) composites

(Matrix : Al, Vf=35%)

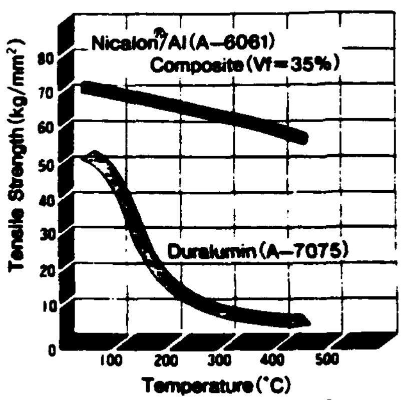
Item		0°	90°	0°±90°
Tensile Strength	(kg/mm^2)	82	7.3	30
Tensile Modulus	(10^3 kg/mm^2)	9.7	6.3	7.7
Flexural Strength	(kg/mm^2)	85	–	–
Flexural Modulus	(10^3 kg/mm^2)	–	–	–
Compressive Strength	(kg/mm^2)	–	–	–
Compressive Modulus	(10^3 kg/mm^2)	–	–	–
I. L. S. S.	(kg/mm^2)	–	–	–
Poisson' s Ratio		0.3	–	–
Coefficient of Themal Expansion	(10^{-6}/°C)	6.9	–	–
Thermal Conductivity	(kcal/m·Hr·°C)	100	–	–
Specific Heat	(J/g°C)	0.93	–	–
Density	(g/cm^3)	2.6	–	–

Properties of Nicalon®/Al(A–6061) composites

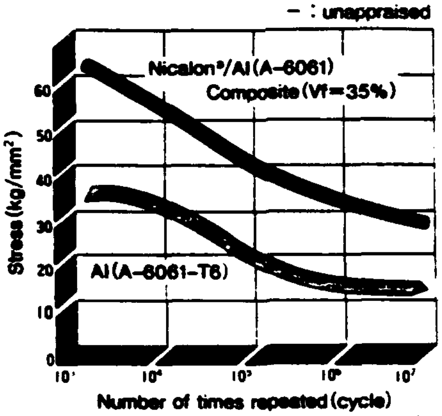
(Matrix : Al, Vf=35%)

Item		0°	90°	0°±90°
Tensile Strength	(kg/mm^2)	71	10.5	20
Tensile Modulus	(10^3 kg/mm^2)	11.5	9.9	11.0
Flexural Strength	(kg/mm^2)	85	19.5	55.6
Flexural Modulus	(10^3 kg/mm^2)	11.0	7.0	8.7
Compressive Strength	(kg/mm^2)	140	23	–
Compressive Modulus	(10^3 kg/mm^2)	15.4	11.5	–
I. L. S. S.	(kg/mm^2)	10.3	2.2	–
Poisson' s Ratio		0.28	0.25	–
Coefficient of Themal Expansion	(10^{-6}/°C)	–	–	–
Thermal Conductivity	(kcal/m·Hr·°C)	–	–	–
Specific Heat	(J/g°C)	–	–	–
Density	(g/cm^3)	2.6	–	–

– : unappraised



Tensile properties of Nicalon®/Al (6061) at High Temperature (Unidirectionally reinforced)



Flexural fatigue properties of Nicalon®/Al (A–6061) (Unidirectionally reinforced)

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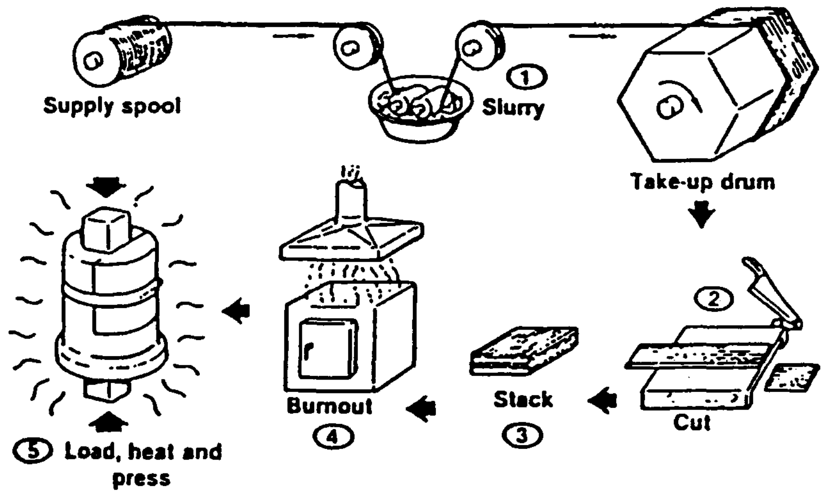
Figure 46



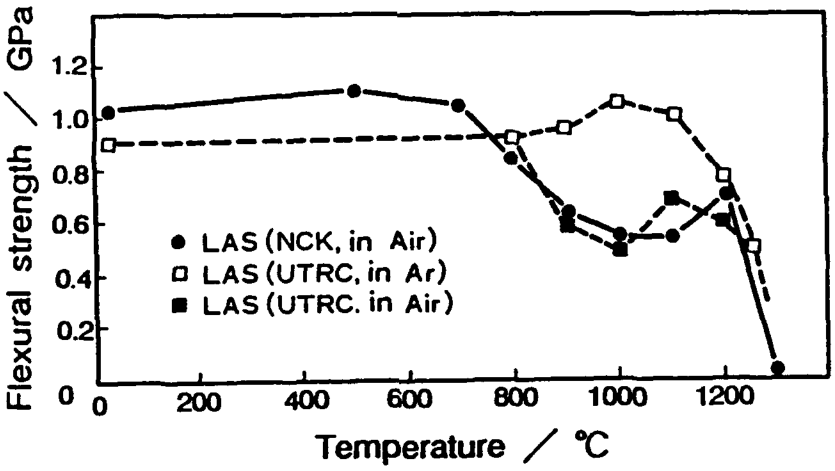
Steps in tape lay-up processing of glass matrix composites.

Figure 47

Table 44

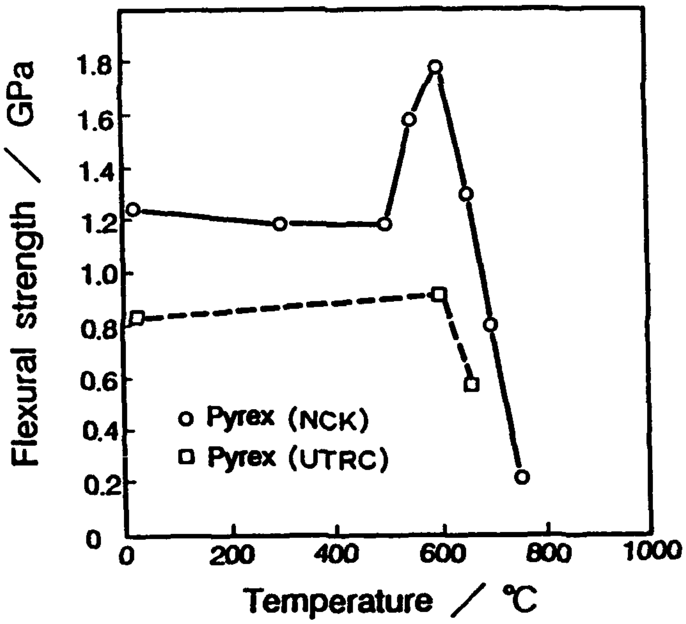
Properties of NICALON-Reinforced Glass and Glass-ceramics

Property		Unit	Pyrex #7740 (Borosilicate Glass)	LAS #4011B (Lithium Alminosilicate)
Fiber Orientation			Uni Directional	Uni Directional
Volume Fraction		%	45	40
Density		g/cm³	2.35	2.50
Flexural Strength	0°, RT	kg/mm²	120	100
	0°, 600°C	kg/mm²	175	100
	0°, 1200°C	kg/mm²	---	60
Frexural Modulus	0°	ton/mm²	12	12
Tensile Strength	0°	kg/mm²	65	55
Tensile Modulus	0°	ton/mm²	13	13
Fracture Toughness		MN/m³/²	23	
Poisson's Ratio			0.19	0.22
Thermal Expansion		$x10^{-6}/°C$	3.2	2.2



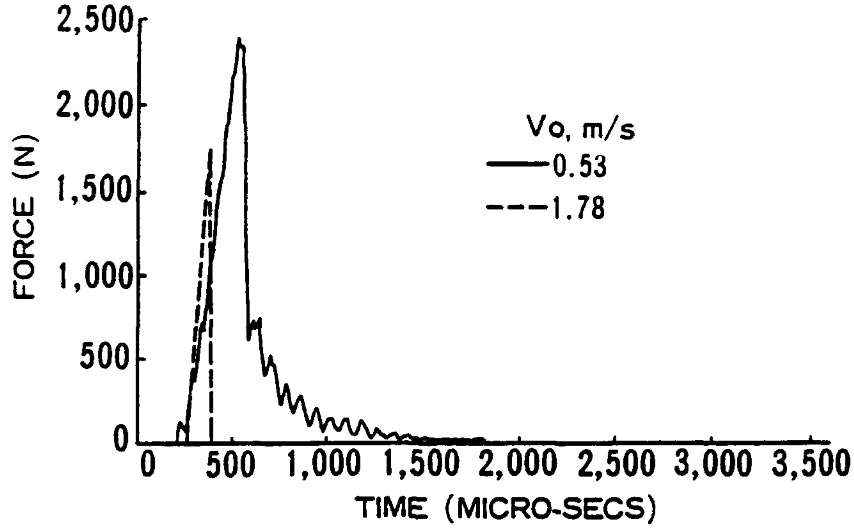
High temperature flexural strength
of NICALON / LAS composite

Figure 48

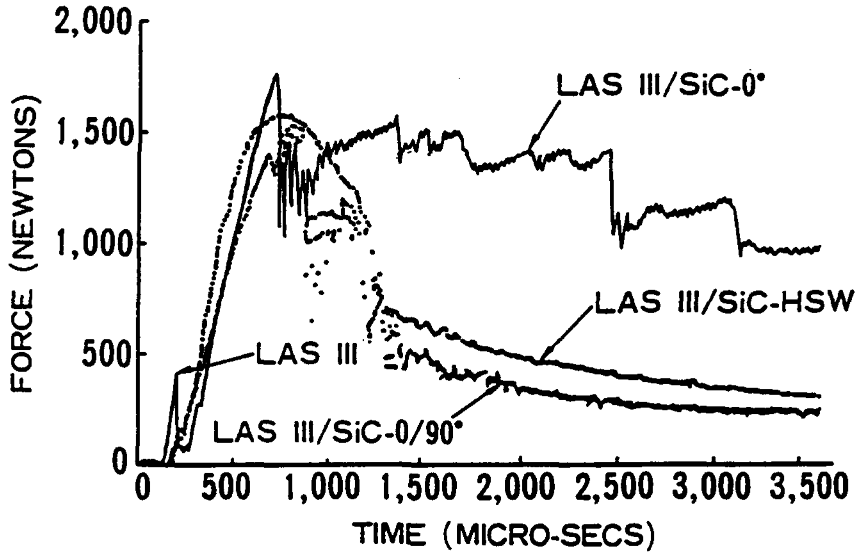


High temperature flexural strength
of NICALON / Pyrex composite

Figure 49



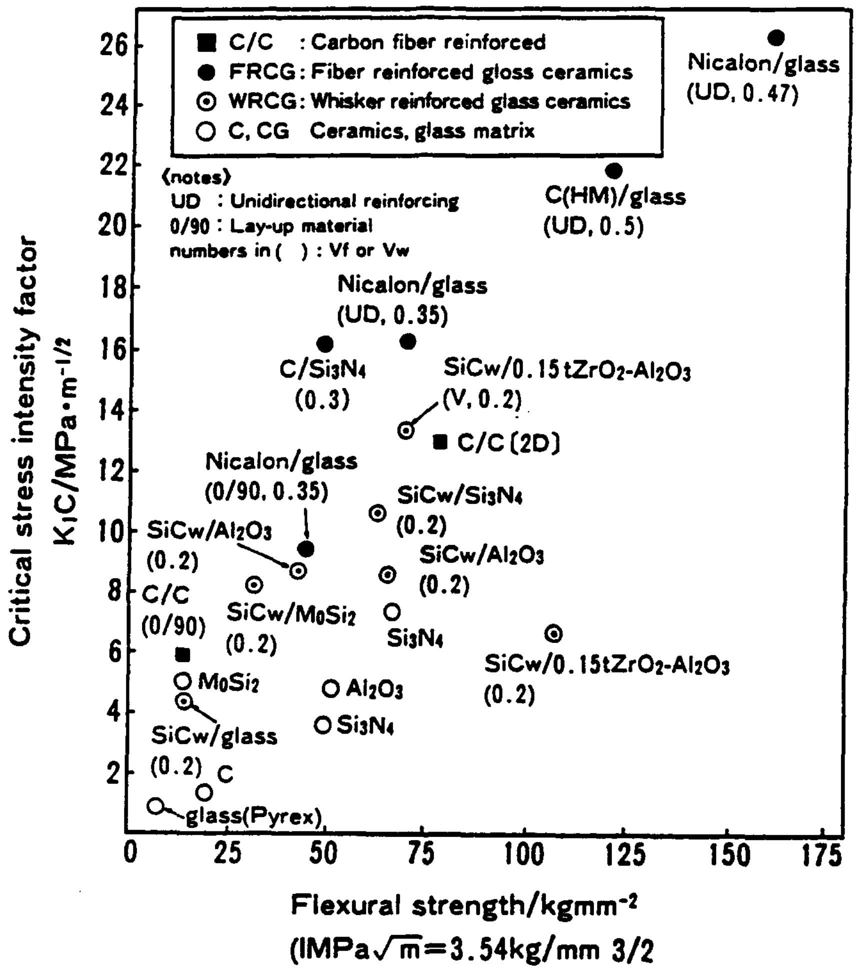
(A) BOROSILICATE GLASS/50 v/o GRAPHITE FIBERS-0/90°



(B) LAS III AND LAS III/44 v/o SiC FIBERS VARIOUS ARCHITECTURES

Impact load-time curves of NICALON reinforced glass ceramics

Figure 50



K_1C and strength of CMCs and ceramics

Figure 51

NICALOCERAM

PROCEDURE OF PRODUCTION

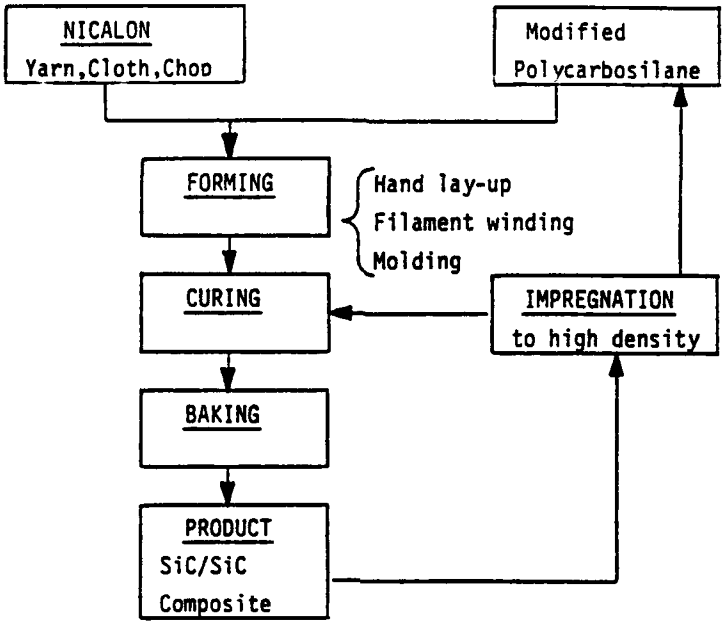


Figure 52

Table 45

Typical properties of "NICALOCERAM"
(NICALON®/SiC composite)

Density (kg/m^3)	1.8×10^3
Fiber volume fraction (%)	30
Flexural strength (MPa)	110
Flexural modulus (GPa)	40
Coefficient of thermal expansion (K^{-1})	3.6×10^{-6}
Thermal conductivity ($Wm^{-1}K^{-1}$)	0.56 (at 773K) 0.73 (at 973K)
Specific heat ($Jkg^{-1}K^{-1}$)	1130 (at 773K) 1170 (at 973K)

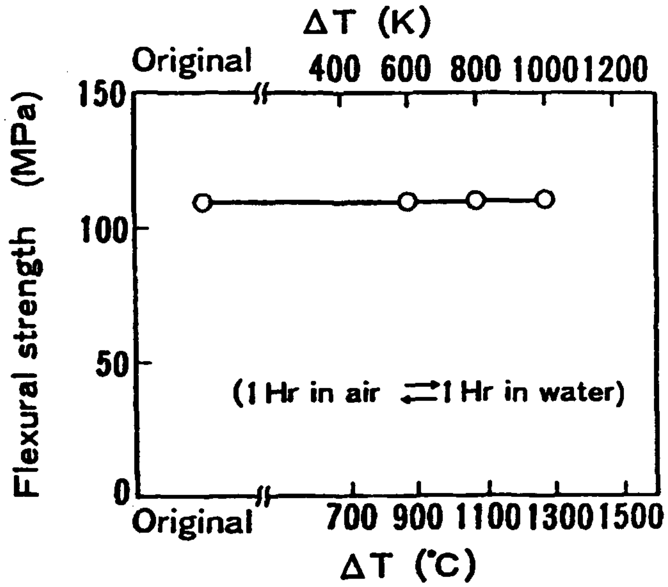


Fig. Flexural strength of "NICALOCERAM" (NICALON®/SiC composite) after elevated temperature exposure

Figure 53

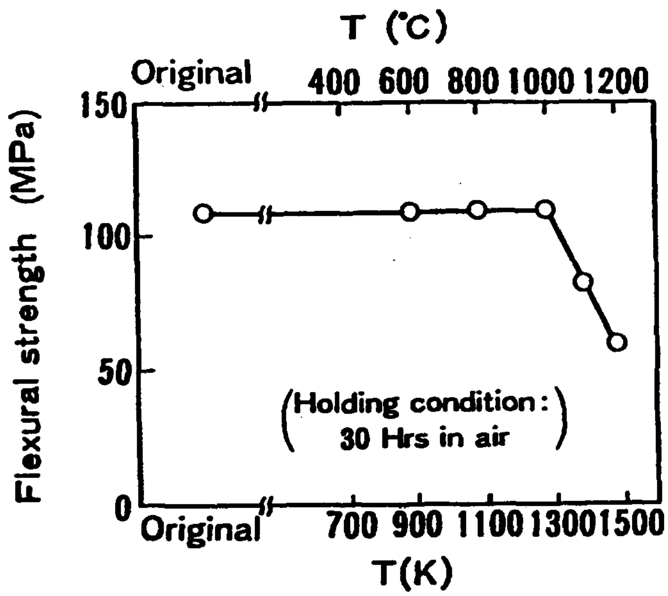


Fig. Flexural strength of NICALOCERAM (NICALON®/SiC composite) after elevated temperature exposure

Figure 54

Properties of Nicalon® Reinforced Glass and Glass-Ceramics Composites

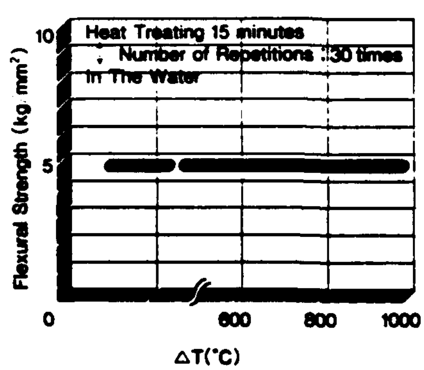
(Vf = 50%) Uni Directional

Item		LAS	Borosilicate
Density	(g/cm^3)	2.45	2.35
Flexural Strength	(kg/mm^2)		
Room Temperature		100	120
600°C (in air)		100	175
1200°C (in air)		60	–
Flexural Modulus		13(R.T.~1200°C)	12(R.T.~600°C)
Fracture Toughness	$(MNm^{-3/2})$		
Room Temperature		20	23
High Temperture in air		22 (1000°C)	24 (500°C)
Coefficient of Thermal Expansion $(10^{-6}/°C)$		3.1	3.2
Thermal Conductivity	(kcal/m·Hr·°C)	1.12 (800°C)	1.16 (600°C)
	(W/mK)	1.30 (800°C)	1.35 (600°C)

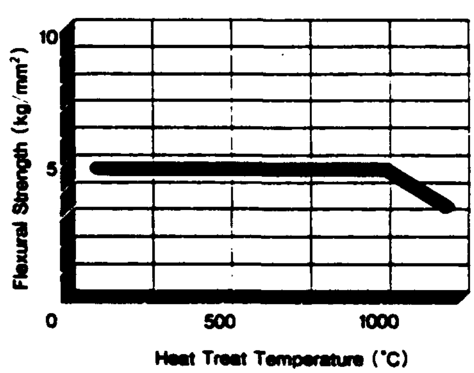
Properties of Nicaloceram (Nicalon®/SiC composite)

(Reinforcement : 12 Harness satin · Vf=30%)

Item		
Density (g/cm^3)		1.8
Flexural Strength (kg/mm^2)	Room Temperature	4.5
Flexural Modulus $(10^3 kg/mm^2)$	Room Temperature	3.5
Coefficient of Thermal Expansion $(10^{-6}/°C)$		3.1
Thermal Conductivity (kcal/m·Hr·°C)	500°C	0.48
	700°C	0.63
Specific Heat (cal/g·°C)	500°C	0.27
	700°C	0.28



Flexural Strength of Nicaloceram After Thermal cycle Test



Flexural Strength of Nicaloceram After Heat Treatment in Air (30Hr)

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Figure 55

5. Carbon-Carbon Composites

NCK produces four grades of C-C; their construction and properties are listed in Table 46. There was passing mention of Nicalocoat to provide oxidation resistance; presumably this is a coating of modified polycarbosilane that can be converted to SiC.

6. Plant Tour

In the Nicalon operation the TAT was shown the precursor polymer in barrels as received from Shinetsu Chemical, the two 250-filament spinning lines (but not the spinning heads or green fiber takeup), and the end of the pyrolysis step and the final takeup. Process details, being proprietary, were sketchy. The TAT also saw graphite powder being compressed into thin sheets to be used in making gaskets, and plasma spraying of Al onto Nicalon fibers (on a drum), followed by hot rolling into MMC sheets. The aluminum preform wire was also observed. Here the Nicalon fiber was desized (by heat) and run through a molten Al bath.

7. Summary

Having been constructed ir 1938, the NCK facilities were the oldest and most rudimentary of all those seen by the TAT. Nevertheless, NCK had a most impressive array of high-technology products and data sheets to demonstrate its dedication and industry.

Table 46

Typical Properties of C/C composite

[CCM代表特性]

Nippon Carbon Co.

	品種	CCM-190B[1]	CCM-190C[1]	CCM-290B[2]	CCM-290C[2]
Heat Treatment temperature	処理温度 ℃	1000	2000	1000	2000
Density	かさ比重	1.6	1.6	1.35	1.35
Bending Strength	曲げ強さ kg/mm²	20.0	16.0	8.0	7.0
Bending modulus	曲げ弾性率 x10³kg/mm²	6.0	6.0	1.5	1.5
Tensile strength	引張り強さ kg/mm²	16.0	15.0	5.0	4.0
Tensile modulus	引張り弾性率 x10³kg/mm²	6.0	6.0	1.5	1.5
Compressive strength	圧縮強度 kg/mm²		13.0		
Shore Hardness	硬さ (ショア硬度)	(⊥) 80~95 [4]	(⊥) 60~85	(⊥) 50~70	(⊥) 45~65
Specific resistivity	固有抵抗 x10⁻³Ω·cm	(∥) 3.5	(∥) 2.0	(∥) 7	(∥) 3.7
Coefficient of Thermal Expansion	熱膨張係数[3] x10⁻⁶/℃	(∥) 0.3 (⊥) 4.0	(∥) 0.8 (⊥) 4.4		
Thermal Conductivity	熱伝導率 kcal/mhr℃		(∥) 5.5 (⊥) 0.5		
Specific Heat	比熱 cal/g℃	0.2	0.2	0.2	0.2

1) 高強度グレード炭素繊維使用 1) High strength Carbon Fiber 1987.12.10

2) 汎用グレード炭素繊維使用 2) General grade Carbon Fiber

3) RT~800℃平均熱膨張率 3) average CTE

4) (∥)繊維に平行方向、(⊥)繊維に直角方向

4) (∥) with fiber (⊥) across fiber

CCM-○○○○

Fiber
1 : High strength Carbon Fiber
2 : General grade Carbon Fiber

→ Base Fiber 90: cross type

→ HTT B: 1000℃ C: 2000℃

J. KEIDANREN

Keidanren, the Japanese Federation of Economic Organizations, is a nonprofit organization representing virtually all branches of economic activities in Japan. It was established in August 1946 through the merger of several economic and industrial organizations active in prewar days. Its officers and committee chairmen are the chairmen or chief executive officers of virtually all major corporations and banks, including Nippon Steel, Toyota, Nissan, Toshiba, Sony, Komatsu, Sumitomo Bank, and Fuji Bank. Hence, its views and recommendations to the Government reflect the consensus of the Japanese business community.

Prior to its arrival in Japan, the TAT was informed by the Mutual Defense Assistance Office (MDO) in the American Embassy of an invitation to address Keidanren. Accordingly, Drs. Wilcox, Minges, and Katz prepared to discuss the TAT mission for DoD and the role of DARPA in defense R&D, and to present overviews of the National Aerospace Plane (NASP) program and the Integrated High-Performance Turbine Engine Technology (IHPTET) initiative as contributors to and users of advanced composites technology.

On the evening of 7 February 1989, Dr. Wilcox and Mr. Bersch, with translator support from the American Embassy, met with Mr. Akihiko Kuno, retired Air Defense Force Major General Naohiko Ohshima, and retired Ground Defense Forces Major General Tsukasa Nishida, of Keidanren's Defense Production Committee, to discuss and plan the agenda and procedures, since all presentations, questions, and answers would require translation.

On 14 February 1989 the TAT met at the New Otani Hotel for a 2-hour session with more than 100 members of the Defense Production Committee. The meeting was run by Dr. Masao Kanamori, Vice Chairman of Keidanren and Chairman of Mitsubishi Heavy Industries, Ltd. A list of the Defense Production Committee appears in Table 47, the list of attendees in Table 48, and the Opening Remarks of Dr. Kanamori are presented in Table 49. After the presentations by Drs. Wilcox, Minges, and Katz, there was a lively question-and-answer session. Discussion items included needs and U.S. facilities for high-temperature materials testing, degree of U.S. R&D emphasis on PMC versus MMC versus CMC, aerodynamic heating demands on NASP materials, the NASP funding profile by organizational category and technology thrust, the oxidation resistance of C-C and SiC and Si_3N_4 matrix composites, future demand for high-performance fibers, and the time frame for IHPTET engine demonstrations.

Table 47

<u>List of Defense Production Committee</u>

(as of ~~Jan.~~ Apr, 1988)

<u>Chairman:</u>

Dr. Masao KANAMORI — Vice Chairman, Keidanren; Chairman, Mitsubishi Heavy Industries, Ltd.

<u>Members:</u>

Asahi Chemical Industry Co., Ltd.

Asahi-Seiki Manufacturing Co., Ltd.

Anritsu Electric Co., Ltd.

Isuzu Motors Limited

C. Itoh & Co., Ltd.

Ishikawajima-Harima Heavy Industries Co., Ltd.

Ishikawa Seisakusho, Ltd.

Ube Industries, Ltd.

Okura & Co., Ltd.

Oki Electric Industry Company, Ltd.

Kayaba Industry Co., Ltd.

Kawasaki Heavy Industries, Ltd.

Kyosan Electric Mfg. Co., Ltd.

Cosmo Oil Co., Ltd.

Komatsu Ltd.

Kobe Steel, Ltd.

Kokusai Electric Co., Ltd.

Sasebo Heavy Industries Company, Limited

Shimazu Corporation

Showa Shell Oil K.K.

Showa Aircraft Industry Co., Ltd.

Shinko Electric Co., Ltd.

Shin Meiwa Industry Co., Ltd.

New Japan Aircraft Co., Ltd.

Nippon Steel Corporation

Sumitomo Chemical Co., Ltd.

Sumitomo Corporation

Sumitomo Precision Products Co., Ltd.

Sumitomo Heavy Industries, Ltd.

Sumitomo Electric Industries, Ltd.

Sony Corporation

Daikin Industries, Ltd.

Daicel Chemical Industries, Ltd.

The Dai-ichi Kangyo Bank, Ltd.

Teijin Seiki Co., Ltd.

Toa Nenryo Kogyo K.K.

Tokyo Keiki Co., Ltd.

Tokyo Aircraft Instrument Co., Ltd.

Toshiba Corporation

Table 47 (Cont'd)

-3-	-4-
Toyo Communication Equipment Co., Ltd.	Fuji Electric Co., Ltd.
Nissan Motor Co., Ltd.	The Furukawa Electric Co., Ltd.
Nissho Iwai Corporation	Howa Machinery, Ltd.
Nippon Aviotronics Co., Ltd.	Matsushita Electric Industrial Co., Ltd.
Nippon Light Metal Co., Ltd.	Marubeni Corporation
Nikon Corporation ~~Nippon Kogaku K.K.~~	Mitsui Engineering & Shipbuilding Co., Ltd.
Nippon Kokan K.K.	Mitsui & Co., Ltd.
Nippon Koki Co., Ltd.	Mitsubishi Chemical Industries Limited
The Industrial Bank of Japan, Ltd.	Mitsubishi Heavy Industries, Ltd.
Japan Aviation Electronics Industry, Ltd.	Mitsubishi Corporation
The Japan Steel Works, Ltd.	Mitsubishi Steel Mfg. Co., Ltd.
Nippon Oil Company, Ltd.	Mitsubishi Oil Co., Ltd.
Nippon Petroleum Refining Company, Limited	Mitsubishi Electric Corporation
NEC Corporation	Mi~~r~~tsubishi Precision Co., Ltd.
Nippon Electronic Co., Ltd.	Mitsubishi Rayon Co., Ltd.
Japan Storage Battery Co., Ltd.	Yuasa Battery Co., Ltd.
Japan Aircraft Mfg. Co., Ltd.	Yokokawa Electric Works, Ltd.
Japan Radio Co., Ltd.	The Yokohama Rubber Co., Ltd.
Nippon Oil & Fats Co., Ltd.	
Hitachi Ltd.	
Hitachi Zosen Corporation	
The Fuji Bank, Limited	
Fuji Heavy Industries Ltd.	
Fujitsu Limited	

Table 48

<u>List of KEIDANREN Participants</u>

(as of February 13, 1989)

February 14, 1989
<at Room Hagi, Hotel New Otani>

Mr. Masao Kanamori	Chairmanirman Defense Production Committee Keidanren Vice Chairman Keidanren Chairman Mitsubishi Heavy Industries, Ltd.
Mr. Yoshio Matsumoto	Manager Composite Material Div. Asahi Chemical Industry Co., Ltd.
Dr. Osamu Nagano	Advisor Ishikawajima-Harima Heavy Industries Co., Ltd.
Mr. Takeo Yamakawa	Advisor Ishikawajima-Harima Heavy Industries Co., Ltd.
Mr. Kazuo Sezaki	Chief Engineer Technical Development Ishikawajima-Harima Heavy Industries Co., Ltd.
Mr. Kanji Murashima	Division Director Aero-Engine & Space Operations Research & Engineering Div. Ishikawajima-Harima Heavy Industries Co., Ltd.
Mr. Yoshihiro Niwa	Section Manager Ishikawajima-Harima Heavy Industries Co., Ltd.

-1-

Mr. Yuji Takemura	Manager Business Planning Dept. Ishikawajima-Harima Heavy Industries Co., Ltd.
Dr. Satoshi Yashima	Manager Aero-Engine Div. Ishikawajima-Harima Heavy Industries Co., Ltd.
Mr. Yoshiyuki Haraguchi	Ishikawajima-Harima Heavy Industries Co., Ltd.
Mr. Takehisa Kaneko	General Manager Direct Sales Dept. Isuzu Motors Ltd.
Mr. Katsuji Takanami	General Manager Deputy Executive in Charge Research & Development Center Isuzu Motors Ltd.
Mr. Yasuo Mikami	Manager Material Div. Research & Development Center Isuzu Motors Ltd.
Mr. Kenji Takagi	Quasi-Director Domestic Sales Dept. Isuzu Motors Ltd.
Dr. Masaru Chikada	Senior Advisor C. Itoh & Co., Ltd.
Mr. Norry Dogan	Aerospace Dept. C. Itoh & Co., Ltd.
Mr. S. Iida	Director C. Itoh Aviation Co., Ltd.
Mr. A. Yamada	Director C. Itoh Aviation Co., Ltd.
Mr. Hosaku Sato	Manager Oki Electric Industry Co., Ltd.

-2-

Table 48 (Cont'd)

Mr. Daigo Kanai	Assistant Manager Aircraft Comp. Div. 2nd Design Section Kayaba Industry Co., Ltd.
Mr. Akira Tsuji	Associate Director General Manager Kawasaki Heavy Industries, Ltd.
Mr. Tamaji Ikeda	Senior Manager Technology Development Group Kawasaki Heavy Industries, Ltd.
Mr. Koji Ito	Senior Manager Material Engineering Section Kawasaki Heavy Industries, Ltd.
Mr. Koumei Kawaji	Manager Kawasaki Heavy Industries, Ltd.
Mr. Shigetoshi Miyazawa	Assistant Manager Kawasaki Heavy Industries, Ltd.
Mr. K. Ohwada	General Manager Kobe Steel, Ltd.
Mr. S. Suzuki	General Manager Kobe Steel, Ltd.
Mr. Morihiko Sugino	Manager New Business Development Dept. Kobe Steel, Ltd.
Dr. T. Mizoguchi	Senior Researcher Kobe Steel, Ltd.
Dr. Akira Kiuchi	Senior Researcher Kobe Steel, Ltd.
Mr. Masahiro Yamasaki	Research Engineer Komatsu Ltd.
Mr. Kan-ichi Sato	Project Manager Komatsu Ltd.
Mr. Junsei Nagai	Chief Engineer Shin Meiwa Industry Co., Ltd.

Mr. T. Ikuyama	Senior Engineer Shin Meiwa Industry Co., Ltd.
Mr. Masaki Kamoshita	Director Products Planning & Research Dept. Showa Aircraft Industry Co., Ltd.
Mr. Hiroshi Tateishi	Simadzu Corporation
Dr. Toru Yukawa	Managing Director Shinko Research Co., Ltd.
Mr. Keiichi Otsuka	Assistant Chief Engineer Shinko Electric Co., Ltd.
Mr. Hiroshi Minohara	General Manager Development Dept. Sumitomo Heavy Industry Co., Ltd.
Mr. Tadao Suzuki	General Manager Sumitomo Heavy Industry Co., Ltd.
Mr. Shigeyuki Mizuho	General Manager Sumitomo Heavy Industry Co., Ltd.
Mr. Ryotaro Hamano	General Manager Defense Industries Coordination Dept. Sumitomo Heavy Industry Co., Ltd.
Mr. Mitsuaki Yoshikawa	Senior Engineer Hiratsuka Research Laboratory Sumitomo Heavy Industry Co., Ltd.
Dr. Toshiyuki Uegaki	General Manager Defense Project Developing Dept. Sumitomo Electric Ltd.
Mr. Tsuneichi Nakano	Senior Manager Hybrid Products Tokyo sales Dept. Sumitomo Electric Ltd.
Dr. Nobumichi Ohya	Chief Engineer Sumitomo Electric Ltd.
Mr. Kiyokazu Oyama	Adviser Sumitomo Corporation

Table 48 (Cont'd)

Mr. Takao Yajima	Aerospace & Defense Section No. 1 Sumitomo Corporation
Mr. Ryuichi Yamayoshi	Manager R&D Corporate Planning Group Sony Corporaiton
Mr. Kazuaki Kumagai	Sales Dept. Ammunition Div. Daikin Industries Co., Ltd.
Mr. Motoh Mizushima	General Manager Industry Research Office The Dai-ichi Kangyo Bank, Ltd.
Mr. Michimasa Nagatani	Manager Lube Product Dept. Toa Nenryo Kogyo K.K.
Mr. Shigeru Fukuda	Senior Manager Defense Products Dept. C^3I Program Div. Toshiba Corporaiton
Mr. Masao Kataoda	GM Group Member Komukai Works Toshiba Corporation
Dr. Haruyoshi Kuno	Technical Director Toshiba Corporation
Mr. Hisahiro Tanaka	Senior Specialist Toshiba Corporation
Mr. Tateo Hosomura	General Manager Aerospace Div. Nissan Motor Co., Ltd.
Mr. Junichi Iwamura	Manager Aircraft Equipment Export Section Nissho Iwai Corporaiton
Mr. Hideaki Mogi	Deputy Manager Aerospace-Proj. Nissho Iwai Corporaiton

Mr. Yoshio Kawamoto	Aircraft Equipment Export Nissho Iwai Corporation
Mr. Shinichi Hosaka	Engineering Manager Engineering Support Dept. Nippon Avionics Co., Ltd.
Mr. Kei-ichi Hara	Manager Ceramics Dept. NKK Corporaiton
Dr. Masanori Kawahara	Principal Research Engineer Polymers Research Dept. Advanced Tech. Research Center NKK Corporaiton
Mr. Kosuke Nakamura	The Industrial Bank of Japan
Mr. Tsuneo Yamaguchi	Senior Managing Director The Japan Steel Works, Ltd.
Dr. Shinichi Nakada	Deputy General Manager Tokyo Research Laboratory The Japan Steel Works, Ltd.
Mr. Kazumi Takahashi	Ordnance Systems Development Section The Japan Steel Works, Ltd.
Dr. Yoshikuni Murakami	Manager New Product Promotion Section Ground Defense Dept. The Japan Steel Works, Ltd.
Mr. Kimihiko Satake	Senior Program Manager NEC Corporation
Mr. Isamu Watanabe	Supervisor Production Engineering Dept. Radio Application Div. NEC Corporation
Mr. Masanao Horie	Director & General Manager Yokohama Plant Japan Aircraft Mfg. Co., Ltd.

Table 48 (Cont'd)

Mr. Shoichiro Sakamoto	Senior Staff Officer Engineering Dept. Japan Aircraft Mfg. Co., Ltd.
Mr. Minoru Yasuda	Assistant Manager General Technical Dept. Nippon Oil & Fats Co., Ltd.
Mr. Motoi Utsunomiya	Director Business Dept. The Shipbuilders' Association of Japan
Mr. Fumito Shinomiya	Chief Engineer Defense Div. Hitachi Ltd.
Mr. Masataka Owada	Senior Engineer Defense Div. Hitachi Ltd.
Mr. Michio Kunikata	Senior Engineer Hitachi Works Hitachi Ltd.
Mr. Takeshi Utsu	Technical Department Manager Inorganic Chemical Products Div. Hitachi Chemical Co., Ltd.
Mr. Hiroaki Hayano	General Manager Planning Dept. Defense Headquarters Hitachi Zosen Corporaiton
Mr. Ryohei Negishi	Defense Equipment Development Dept. Defense Headquarters Hitachi Zosen Corporaiton
Mr. Keiichi Matsumura	Senior Research Engineer Fuji Electric Co., ltd.
Mr. Wataru Yamagishi	Senior Engineer Inorganic Materials Laboratory Fujitsu Laboratories Ltd.
Dr. Seiya Ogawa	General Manager Materials Div. Fujitsu Laboratories Ltd.

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Mr. Yasumasa Honda	Managing Director General Manager, Aircraft Div. Fuji Heavy Industries Ltd.
Mr. Satoshi Idei	General Manager Market Development Aircraft Div. Fuji Heavy Industries Ltd.
Mr. Toshio Sakakibara	Manager Material Research Section Research & Laboratories Dept. Aircraft Engineering Div. Fuji Heavy Industries Ltd.
Mr. Hiroyuki Nakatsubo	Deputy General Manager Advanced Projects Office Aircraft Engineering Div. Fuji Heavy Industries Ltd.
Mr. Yoshio Tanaka	Senior Staff Manager General Planning Dept. R & D Div. The Furukawa Electric Co., Ltd.
Mr. H. Satoh	Adviser Matsushita Electric Industrial Co., Ltd.
Mr. Hirotada Yaginuma	Matsushita Electric Industrial Co., Ltd.
Mr. Norio Okamoto	General Manager Defense Equipment Section II Marubeni Corporation
Dr. Seiichiro Nishihara	Manager Engineering Dept. Mitsui Engineering & Shipbuilding Co., Ltd.
Mr. Teiichi Yamazaki	Manager Advanced Materials and Products Div. Mitsui Engineering & Shipbuilding Co., Ltd.
Mr. Koichiro Nomura	Project Manager Mitsubishi Heavy Industries, Ltd.
Mr. Hidetaka Ichikawa	Manager Mitsubishi Heavy Industries, Ltd.

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Table 48 (Cont'd)

Mr. Toyoji Ogino	Manager Sales Group Special Vehicle Dept. Mitsubishi Heavy Industries, Ltd.
Mr. Tamon Ikeda	Manager Structure Designing Section Aircraft Engineering Dept. Aircraft & Special Vehicle Headquarters Nagoya Aircraft Works Mitsubishi Heavy Industries, Ltd.
Mr. H. Ueda	Manager Planning & Administration Dept. Mitsubishi Heavy Industries, Ltd.
Mr. Mitsuru Higaki	Deputy Manager Planning & Administration Dept. Aircraft & Special Vehicle Headquarters Mitsubishi Heavy Industries, Ltd.
Mr. Takashige Koga	Senior Engineer Defenseship Dept. Nagasaki Shipyard & Machinery Works Mitsubishi Heavy Industries, Ltd.
Mr. Hiroyuki Migita	Engineer Defense Ship Dept. Nagasaki Shipyard & Machinery Works Mitsubishi Heavy Industries, Ltd.
Mr. Masafumi Inoue	Managing Director Mitsubishi Steel MFG. Co., Ltd.
Mr. Minoru Usami	General Manager Mitsubishi Electric Corporation
Mr. Shin Utsunomiya	Manager Engineering Section Metals & Ceramics Dept. Materials & Electronic Devices Laboratory Mitsubishi Electric Corporation
Dr. Ichiro Taniguchi	Manager SAM Systems Dept. Mitsubishi Electric Corporation

Mr. Nobuo Kamei	Mechanical Systems Dept. Aerodynamics and Propulsion Section Mitsubishi Electric Corporation
Mr. Eiji Kodama	Manager Space Electronics Des. Section Mitsubishi Precision Co., Ltd.
Mr. Shuichi Sato	Adviser Yuasa Battery Co., Ltd.
Mr. Ichiro Funakoshi	Advisor The Yokohama Rubber Co., Ltd.
Mr. Hiroshi Morikawa	Secretary General Office of Defense Production Committee Keidanren
Mr. Naohiko Oshima	MAJ. GEN. Air Force (ret) Research Associate Defense Production Committee Keidanren
Mr. Tsukasa Nishida	MAJ. GEN. Army Force (ret) Research Associate Defense Production Committee Keidanren
Mr. Akihito Kuno	Office of Defense Production Committee Keidanren

Table 49

Tentative Draft of Opening & Closing Remarks by Dr. Kanamari, Chairman of Defense Production Committee, KEIDANREN (As of Feb. ~~14~~ 13 1989)

Feb. 14, 1989

Rm. HAGI, Hotel New Otani,

Tokyo, Japan

1. Opening Remarks

My name is Kanamori, Vice Chairman of KEIDANREN and Chairman of the Defense Production Committee, KEIDANREN. I would like to thank Doctors Wilcox, Minges, Katz for joining us in spite of your tight schedules. I would also like to express my gratitude to Captain Dziedzic, Chief , Mr. Hastings, Deputy Chief , Mr. Allen, Director of R & D Exchange, and others of Mutual Defense Assistance Office, Japan.

Let me also thank the members of the Defense Production Committee for participating in spite of your busy schedules.

As you have been informed in advance, the DOD's TAT led by Dr. Wilcox has come to Japan to study our country's composite materials technology under the shcheme of the Japan/U.S. System & Technology Forum between the Defense Agency and the DOD.

We are very much interested in what the DOD including DARPA is doing in the field of leading —edge composite materials science and technology as the most advanced defense R & D country in the West. We are also interested in how the experts who have come to Japan this time regard composite materials R & D in Japan, partly because joint R & D between the U.S. and Japan is talked about as one of the current topics. The large turn—out today of Japanese experts reflects this interet.

Time is limited so let us strart the presentation. Dr. Wilcox, please.

III. SUMMARY OF TAT OBSERVATIONS

This section seeks to provide an overview of high-temperature composite technology in Japan by brief discussions of each type of fiber and composite. There is no intention to repeat the detailed technical information provided in the visit reports in Section II. Rather, the purpose is to guide the reader who is interested in a specific material--say, carbon fibers--to the appropriate visit reports. There is also no intention to provide a comprehensive summary of all related activity under way in Japan; the TAT visited only a limited number of Japanese companies, and this discussion is confined to them. Lastly, no specific comparisons of Japanese and U.S. technologies are intended; U.S. sources and U.S. products are sometimes mentioned for illustrative purposes.

As was noted earlier, high-temperature composite technology in the United States has been mostly a product of Department of Defense (DoD) and National Aeronautics and Space Administration (NASA) support, to advance aerospace technology. In Japan the majority of R&D is supported and carried out by industry, to meet market needs; thus one might expect a lower emphasis on high technology. However, these distinctions are not clear-cut. For example, most development of PAN- and pitch-based C fibers has been industry supported in both the United States and Japan, while ceramic fiber development has received government support in both countries. On the other hand, most R&D work on MMCs, CMCs, and C-C in the United States has had government support, while (until the new MITI thrust for high-temperature composites began) the availability of the Nicalon and Tyranno fibers and the U.S. aerospace interest in MMCs, CMCs, and C-C has invited the Japanese development of their own MMCs and CMCs, especially Tyrannohex (Ube) and Nicaloceram (NCK).

A. CERAMIC AND GLASS FIBERS

The Japanese role in ceramic fibers and much of the continuous-fiber high-temperature composite technology exist today because of the work of the late Professor S. Yajima of Tohoku University throughout the 1970s. He conducted extensive research on the synthesis of organosilicon polymers (polycarbosilanes) that could be spun into fibers and pyrolyzed to ceramic fibers with useful mechanical properties. Eventually he

was awarded more than 40 patents. He transferred some of them to Nippon Carbon in 1975 and, as noted in greater detail in the NCK visit report (Section II-I), the result was Nicalon, a SiC fiber that has been marketed in the United States by Dow Corning since 1982.

In the early 1980s Dr. Yamamura of Ube Industries conducted research under Professor Yajima that led to the synthesis of polytitanocarbosilane. This was pyrolyzed to a SiC fiber containing about 2 per cent Ti. It is called Tyranno and is available in the United States from Textron Specialty Materials.

The market for both of these SiC fibers has been limited to some "high-tech" sporting goods in Japan, but both Nicalon and Tyranno are being used in MMC and CMC research in the United States, Japan, and Europe. The TAT heard differing reports about the high-temperature limits of these fibers, but both appear useful to about 1200°C (in contrast to C fibers, which oxidize at around 700°C, even when in composites, because of cracks, pores, and exposed ends). Details on the product forms and properties for Nicalon and Tyranno are available in the Ube and NCK visit reports (Sections II-A, II-I). Activities at Ube and NCK are seeking improvements in strength, modulus, thermal stability, and ability to tailor electric properties.

More recently Tonen has developed a silicon nitride fiber based on the pyrolysis of a polysilazane polymer precursor. Though the Tonen fiber is not yet in production, small quantities are available for evaluation. Preliminary data suggest an upper temperature limit about 100°C higher than that of the SiC fibers, but the data are very limited.

Shimadzu is conducting research on the Si-Ca-Al-O-N system and drawing oxynitride glass fibers with N contents up to about 8 per cent. Shimadzu reports fiber strength comparable to that of E glass and elastic modulus values more than twice those available in any commercial glass fiber, but the fibers have not reached pilot-scale development. Shimadzu has hopes for a market between S glass and C fibers.

In summary, Japan is the world leader in polymer-derived, small-diameter (about 10 micron) SiC and Si_3N_4 fibers.

B. CARBON FIBERS

The chronology of carbon fiber development from 1958 to 1974, which appears in the Toray visit report (Section II-D), identifies the roles of the United States, the United Kingdom, and Japan in the early stages of C fiber development based on rayon,

PAN, and pitch. In the intervening years the use of rayon as a precursor has almost disappeared (except for use in some rocket nozzles produced in the United States), and PAN-based C fibers have grown to a production capacity (and maybe a market) about seven times that of pitch-based fibers. PAN fibers are used mostly in polymer matrix composites for sporting goods (Japan) and aerospace components (United States and Europe). Between them, Toray and Toho Rayon (not visited) and their overseas affiliates and licensees seem to have about two-thirds of the world production capacity, and the Japanese influence on the market is even greater because Hercules, the largest U.S. producer, uses PAN from Sumitomo Chemical. (However, a recent DoD directive requiring that by 1991 at least 50 per cent of C fibers in U.S. aerospace production must be from U.S.-produced precursor materials is leading to the construction of several PAN-producing facilities in the United States).

PAN-based C fibers from Toray are available over a very wide range of strengths and stiffnesses. In fact, Toray believes it has about reached the physical limits available from C fibers and, because the properties of many of its fibers are not being fully translated into composite properties (in polymer matrix composites), it is focusing its research on improvements in resin matrices and, in particular, on the fiber-matrix interface.

Many Japanese companies have considered the C fiber market, and a number have withdrawn from one or another part. Toray, for instance, has ended its activity in pitch-based fibers to focus solely on PAN-based fibers. KOBELCO studied production of both PAN and pitch fibers but dropped PAN fibers and is now considering building a pilot plant to produce fibers from mesophase pitch derived from coal tar. NCK, after many years in the carbon fiber business, has withdrawn from a partnership with Asahi Chemical that was producing PAN-based fibers known as Carbolon; NCK too plans to concentrate on pitch fibers from mesophase pitch derived from coal tar. At present NCK is producing about 1 ton per month, apparently for in-house consumption.

It is said that PAN-based fibers have most of the C fibers market because they combine lower cost, adequate stiffness for most purposes, and better strength--especially compressive strength--than is available from pitch-based fibers. Nonetheless, Tonen is making a substantial effort to develop and market a petroleum-based mesophase pitch C fiber that can compete with PAN fibers on strength and cost. Tonen says it can already provide high-modulus pitch-based fibers with higher strength and lower cost than the competition.

In general, PAN-based fibers are used mostly in polymeric matrices. Pitch-based C fibers have been used in polymer, metal, and glass matrices, usually when great stiffness and dimensional stability are required. Neither is used much in high-temperature composites because they oxidize at around 700°C, but both are used in C-C composites for very-high-temperature applications where oxidation will not occur or where its effects can be endured.

Overall, Japan has world-class, maybe world-leading, capabilities in PAN- and pitch-based C fibers, and the competition is strong within Japan and throughout the world.

C. WHISKERS

Ube produces α- and β-type silicon nitride whiskers in large quantities by the decomposition of silicon diimide. Ube's whiskers are used with polymer, metal, and ceramic matrices. Data sheets for both types of fibers appear in the Ube visit report in Section II-A.

KOBELCO produces β SiC whiskers by a solid state reaction (vapor-liquid-solid over a Ni catalyst). KOBELCO is currently producing 100 kg/month and is considering a 6-12 tons/year pilot plant. Most current production is used in house in MMC and CMC R&D, but presumably whiskers will be for sale if the pilot plant is built.

D. ADVANCED COMPOSITES

Although the intention of the TAT was to visit production facilities, not R&D laboratories, it appears that the only high-temperature composite component in production in Japan is an MMC piston head used in Toyota diesel engines. Three different suppliers (none on the TAT itinerary) produce these piston heads by squeeze-casting aluminum around a preform of low-cost fibers such as Kaowool or Saffil. Hence all of the following information on MMCs, CMCs, and C-C represents R&D status.

1. Polymer Matrix Composites

As noted in Section I-B, organic polymer matrix composites were excluded from the TAT agenda because of the TAT emphasis on high-temperature performance. However, extensive PMC capabilities were displayed to the TAT at Toray, where the principal emphasis was on PAN-based C fibers and their use in polymer composites, and at MELCO, where many satellite structural components are made from polymer matrix composites.

2. Continuous-Fiber-Reinforced MMCs

The TAT encountered virtually no C fiber MMC. Toray reported to have done some R&D in the 1970s but found no demand or market. IHI mentioned the use of C preforms but gave no details, and MELCO, in a list of publications, included one mention of a short C fiber/Al composite.

Most of the continuous-fiber MMC work that the TAT saw was at the ceramic fiber producers (Ube and NCK) and at the prospective producers/users of high-temperature composite components (Toyota, IHI, and MELCO). In most cases the reinforcement was with Nicalon or Tyranno fiber and, except for a brief mention of Ti and superalloy matrices at IHI, the matrix was always aluminum. Ube in particular mentioned the need for special Al alloys, using Ni or Ti instead of Zn or Mg as alloying elements, to reduce reaction at the fiber surfaces. Both Ube and Toyota emphasized the use of ceramic particles or whiskers between the fibers to assure optimum penetration of the molten Al when doing squeeze casting. Apparently the technique was developed at Toyota, and Ube produces a special Tyranno fiber known as hybrid Tyranno that has ceramic powder or whiskers adhering to the ceramic fibers.

The principal process to produce MMCs seems to be squeeze casting. However, Ube and IHI mentioned preparing sheet prepreg by plasma spraying Al onto ceramic fibers wound on a drum, then cutting and stacking the sheets prior to hot-pressing them. IHI uses the same process with superalloy powder and with metal foil. MELCO, in a list of publications, included at least two papers on fabrication of MMCs by laser processing, but the TAT learned no details. In a summary paper reproduced in the NCK visit report (Section II-I), NCK describes the preparation of a Nicalon/Al wire that is made into MMC by hot-pressing or hot-rolling or by making a preform for die casting.

3. Particulate/Whisker-Reinforced MMCs

Ube representatives mentioned using their α Si_3N_4 whiskers in aluminum matrices, but they gave no further information. They also noted that either Si_3N_4 or SiC whiskers could be used to produce the Hybrid Tyranno used in continuous-fiber MMCs.

Kobelco representatives mentioned using their β SiC whiskers in MMC R&D conducted elsewhere. MELCO listed several papers it had published on whisker-reinforced metals but provided no additional details.

4. Continuous-Fiber CMCs

The TAT found only a little CMC work comparable to that in the United States. In 1984 NCK started R&D on Nicalon-fiber-reinforced glass ceramics similar to UTRC's Compglas; some information on the process and properties appears in the NCK visit report (Section II-I). Toyota CRDL reported no prior work but an intention to start (maybe as part of the new MITI program?), while IHI observed that the available ceramic fibers were limited to 1200°C, apparently implying no interest.

However, the two ceramic fiber producers have developed interesting new processes and products. Ube's Tyrannohex consists of webs of fibers in unidirectional or crossply layups, without any matrix, that are hot-pressed to produce a "ceramic wood" that breaks at a strain of about 3 percent. At the time of the TAT visit the process was new and data were scarce, but the material looked very promising.

NCK's Nicaloceram is a SiC-SiC CMC somewhat similar to SEP's but is produced by a process of the C-C type. A Nicalon fiber preform is impregnated with a modified polycarbosilane (similar to the Nicalon precursor), cured and baked, reimpregnated, etc., until the desired density is achieved. The Nicaloceram property data included in the NCK visit report do not show very high values, but the research was in an early stage.

5. Whisker-Reinforced CMCs

The TAT found only limited activity in whisker-reinforced CMCs and not much enthusiasm. Ube has used up to 30 per cent Si_3N_4 whiskers in α and β Sialons, and reported that β Si_3N_4 whiskers could double the fracture toughness of β Sialon. KOBELCO staff members mentioned using their β SiC whiskers in CMC R&D but provided no further information. Apparently Toyota CRDL has conducted extensive R&D on whisker-reinforced CMCs. CRDL staff members described injection-molding β Si_3N_4 reinforced with SiC whiskers. Several times, however, they expressed curiosity about the U.S. emphasis on reinforcing ceramics with whiskers, claiming to achieve similar properties at much lower cost with monolithic ceramics.

IHI has also looked at SiC whiskers in Si_3N_4 but has found no improvement in properties over those of monolithic ceramics. IHI spokesmen noted that while the whiskers increased costs, the real cost increase was in processing. MELCO has published several papers on its research on SiC whiskers in glass, but a MELCO staff member said, "The climate is not yet right and the properties we have obtained are not attractive."

6. Carbon-Carbon Composites

NCK has been in the carbon/graphite business since 1915 and has been technology oriented throughout that time. However, the TAT did not learn when NCK began producing C-C nor anything about the process or application, mostly because of the focus on Nicalon, MMCs, and CMCs. However, a property sheet describing four grades of C-C was received and is reproduced in the NCK visit report (Section II-I).

KOBELCO reported that it started to study carbon/graphite materials 10 years ago and has focused on C-C in recent years. Its target is aircraft brakes, but they are not yet in production. KOBELCO does produce three grades of C-C, two of them loaded with 30 percent Cu or Al, for bushings, electric motor brushes, etc. A data sheet is provided in Section II-B.

MELCO said it was not working on C-C because it did not have the requisite equipment. IHI was planning to start work on C-C as part of the new MITI program. Nissan is reported to produce C-C for rocket motor nozzles, but the TAT did not visit Nissan.

IV. FINDINGS

The van rides to and from the Japanese companies generally took from 1 to 2.5 hours. During these rides, and at breakfast meetings on the last two mornings in Tokyo, the TAT discussed a variety of technical and general observations. These observations, for which there was a consensus, are reported below as findings.

A. GENERAL FINDINGS

The general findings of the TAT may be summarized as follows:

- The influence of the Japan Defense Agency (JDA) and the Ministry of International Trade and Industry (MITI) helped a great deal in opening doors for the TAT. The 9 Japanese companies (at 10 installations) were, by and large, very open in their discussions and plant tours. In some cases the access to and flow of information was unprecedented.
- Most U.S. R&D in fibers and composites has been for high-performance, often high-temperature, aerospace applications. Most composite research and some fiber research have been supported by the Department of Defense (DoD) and the National Aeronautics and Space Administration (NASA). Any commercial applications followed the Government-sponsored developments. In contrast, Japanese R&D in fibers and composites has largely been company sponsored. Japanese companies have been laying the groundwork--positioning themselves in sporting goods, and preparing for future markets in automotive and, eventually, aerospace applications. Their efforts may be high technology but are not high temperature at present. The use of both Nicalon and Kevlar fibers in the bow of a tennis racket and graphite fibers in the handle illustrates the high-technology interest of the Japanese consumer.
- Japanese materials scientists and engineers are very capable and well trained, very industrious, and very persevering. The Japanese have about 30,000 young scientists and engineers engaged in advanced materials R&D. They often pursue a technology where no payoff is apparent; they believe an application can be found for a good material. The costs of development and time--up to 15 years--are sometimes enormous. Japanese patience and perseverance is impressive.

- Some attrition in materials programs was apparent: (1) At one time about 30 Japanese companies were working on carbon fibers; there are many less now. Toray dropped pitch fibers to focus on PAN fibers. Tonen emphasizes pitch. Nippon Carbon transferred its share of a joint PAN effort to Asahi but continues to produce pitch-based carbon fibers. (2) Several companies have stopped R&D of particulate/whisker ceramic matrix composites (CMCs) in favor of monolithic ceramics; they claim processing costs outweigh any likely improvements in performance.
- Many Japanese companies seek licensing and distribution relationships with U.S. companies: Nippon Carbon distributes Nicalon fibers through Dow Corning, and Ube Industries distributes Tyranno fibers via Textron Specialty Materials, while Toray has cross-licenses on some high-performance resins with Hexcel.
- Japanese pilot plants are BIG: one fiber processing line was at least 250 feet long. The plants are able to wring out the potential of the equipment and the process in a realistic manufacturing setting as well as to find potential problems. In contrast, DoD-supported pilot plants are often "tabletop glassware" operations.
- The Japanese closely follow worldwide developments in composites and other technologies. For example, Keidanren (Japan Federation of Economic Organizations) collected DARPA's Annual Report to Congress for the years 1979-1987, combined and restructured it by technology subject, translated it, and distributed it to Japanese industry.

B. TECHNICAL FINDINGS

The technical findings of the TAT may be summarized as follows:

- Japan is very strong in carbon/graphite fibers. Toray, with PAN-based fibers, and Tonen, with pitch-based fibers, are outstanding. Current emphases are on cost reduction and improvements in resin matrices and fiber-matrix interfaces in order to maximize translation of the fiber properties into the composite.
- Japan appears to lead the world in high-performance ceramic fibers prepared from organosilicon polymeric precursors: Nippon Carbon has Nicalon; Ube Industries has Tyranno; and Tonen seems about ready to market a silicon nitride fiber.
- Fiber production operations are well designed and constructed (e.g., several spinning and curing operations are done in clean rooms). Most operations are highly automated, with minimal manpower required.

- Some preceramic polymers appear to have great potential for additional product applications such as oxidation protection coatings, tapes, films, adhesives, and laminated macrocomposites. Tailored "hybrid" reinforcement schemes seem likely to evolve.

- The TAT encountered considerable activity in metal matrix composites (MMCs), but mostly with aluminum as the matrix and short fibers as the reinforcement. Squeeze casting is well developed and seems to be the preferred fabrication technique. Toyota is producing MMC in piston heads for diesel engines, but the TAT saw no indication of commodity MMC forms such as Dural in the United States is said to produce.

- The TAT encountered almost no activity in high-temperature MMCs, that is, almost nothing in titanium or intermetallic matrices. However, this observation requires two qualifications. First, the TAT did not visit Fuji, Kawasaki, or Mitsubishi Heavy Industries, all known to be active in all types of composites. Second, just before the TAT arrived in Japan, the Japanese Diet approved a new MITI Advanced Composite Materials Program ($80 million from the government over 8 years; additional funds from participating industry are sometimes 10 times greater than the government money) to start in April 1989, with emphasis on high-temperature composite applications for aerospace and the Japanese spaceplane, HOPE, which is to be designed in 1992. Virtually all of the companies the TAT visited expressed great enthusiasm for their respective roles in the program.

- The interest and activity of the Japanese in continuous-fiber CMCs appears to have been limited because they saw no market. Promising efforts, such as Ube's Tyrannohex, Nippon Carbon's Nicaloceram and LAS materials, and Tonen's Si_3N_4 matrix composites, were all in early stage of development at modest effort levels. This is likely to change with the new MITI initiative described above.

- Japanese interest in particulate/whisker-reinforced CMCs seemed to be waning. Several companies said that processing costs were prohibitive and that the mechanical properties of monolithic ceramics were comparable to those of discontinuously reinforced ceramics. The TAT saw several instances of monolithic ceramics, especially silicon nitride, being used in automotive turbocharger rotors.

- Just as in the United States, there is a very poor data base on the performance of ceramic and graphite fibers in MMCs and CMCs. There is a particular need for more information on the present capability and thermal stability of ceramic fibers at high temperatures.

- The Japanese are accelerating their research in C-C, especially in the new MITI program. Besides C-C work observed by the TAT at Kobe Steel and Nippon

Carbon, it was reported that Mitsubishi Heavy Industries and the Space Division of Nissan Motors produce C-C components for rocket engines. Expanded applications in aircraft brakes are expected soon. The TAT saw no signs of work for gas-turbine-engine or reentry applications. However, promising developments in fibers and mesophase pitch suggest rapid growth in the MITI program.

APPENDIX A

BASIS FOR JAPAN-U.S. TRANSFER OF MILITARY TECHNOLOGIES*

CONTENTS

* Excerpted from *Defense of Japan*, a white paper published annually by the Defense Agency, Japan, and translated by the Japan Times, Ltd.

Mutual Defense Assistance Agreement Between Japan and the United States of America (Excerpts)

Entered into force, May 1, 1954
Treaty No. 6

ARTICLE I

1. Each Government, consistently with the principle that economic stability is essential to international peace and security, will make available to the other and to such other governments as the two Governments signatory to the present Agreement may in each case agree upon, such equipment, materials, services, or other assistance as the Government furnishing such assistance may authorize, in accordance with such detailed arrangements as may be made between them. The furnishing and use of any such assistance as may be authorized by either Government shall be consistent with the Charter of the United Nations. Such assistance as may be made available by the Government of the United States of America pursuant to the present Agreement will be furnished under those provisions, and subject to all of those terms, conditions and termination provisions of the Mutual Defense Assistance Act of 1949, the Mutual Security Act of 1951, acts amendatory and supplementary thereto, and appropriation acts thereunder which may affect the furnishing of such assistance.

2. Each Government will make effective use of assistance received pursuant to the present Agreement for the purposes of promoting peace and security in a manner that is satisfactory to both Governments, and neither Government, without the prior consent of the other, will devote such assistance to any other purpose.

3. Each Government will offer for return to the other, in accordance with terms, conditions and procedures mutually agreed upon, equipment or materials furnished under the present Agreement, except equipment and materials furnished on terms requiring reimbursement, and no longer required for the purposes for which it was originally made available.

4. In the interest of common security, each Government undertakes not to transfer to any person not an officer or agent of such Government, or to any other government, title to or possession of any equipment, materials, or services received pursuant to the present Agreement, without the prior consent of the Government which furnished such assistance.

Statement of Chief Cabinet Secretary on Transfer of Military Technologies to the United States

January 14, 1983

Since June 1981, the Japanese Government has received requests from the U.S. Government for exchange of defense-related technologies. After careful studies on the transfer to the U.S. of "military technologies" as a part of such exchange, the Japanese Government has reached the following conclusion, which was approved by the Cabinet meeting today:

1. Under the Japan-U.S. security arrangements, the U.S. and Japan, in cooperation with each other, are to maintain and develop their respective capacities to resist armed attack. In improving its defense capacities, Japan has been benefiting from various kinds of cooperation extended by the U.S., including transfer of U.S. technologies to Japan. In view of the new situation which has been brought about by, among other things, the recent advance of technology in Japan, it has become extremely important for Japan to reciprocate in the exchange of defense-related technologies in order to ensure the effective operation of the Japan-U.S. Security Treaty and its related arrangements, which provide for and envisage mutual cooperation between Japan and the U.S. in the field of defense, and contributes to peace and security of Japan and in the Far East.

2. The Japanese Government has so far dealt with the question of arms export (including transfer of "military technologies") in accordance with the Three Principles on Arms Export and the Government Policy Guideline on Arms Export). In view of the foregoing, however, the Japanese Government has decided to respond positively to the U.S. request for exchange of defense-related technologies and to open a way for the transfer to the U.S. of "military technologies" (including arms which are necessary to make such transfer effective) as a part of the technology exchange with the U.S. mentioned above; such transfer of "military technologies" will not be subject to the Three Principles on Arms Export. The implementation of such transfer will be made within the framework of the relevant provisions of the MDA Agreement. In this manner, the fundamental objective of refraining from aggravating international disputes, which Japan upholds as a nation committed to peace and on which the Three Principles are based, will be secured.

3. The Japanese Government will continue to maintain, basically, the Three Principles on Arms Export and to respect the spirit of the Diet Resolution on arms export adopted in March 1981.

Outline of Exchange of Notes on Transfer of Military Technologies to the United States Signed Nov. 8, 1983

1. Subject to the detailed arrangements to be concluded for implementing the present understanding, the Government of Japan will authorize, in accordance with the relevant laws and regulations of Japan, transfer to the Government of the United States of America and the the persons authorized by it of such military technologies necessary to enhance defense capability of the United States of America, as will be identified and determined in accordance with the provisions of paragraph 2 below. (Paragraph 1)

2. A Joint Military Technology Commission (hereinafter referred to as "the JMTC") shall be estabished as the means for consultation between the Government of Japan and the Government of the United States of America. Based on the information received from the United States Section and discussion within the JMTC, the Japanese Section shall determine such military technologies as are appropriate to be authorized by the Government of Japan for transfer. (Paragraph 2)

3. The detailed arrangements will be concluded between the competent authorities of the two Governments in order to implement the present understanding. (Paragraph 3)

4. The present understanding will be implemented in accordance with the Japan-U.S. Mutual Defense Assistance Agreement which prohibits (a) the furnishing and use of any assistance which is inconsistent with the charter of the United Nations. (b) the use of any assistance for purpose other than those specified and (c) the transfer of any equipment, materials or services without prior consent of the Government which furnished such assistance. (Paragraph 4)

5. The Government of the United States of America agrees to take such security measures as would guarantee the same degree of security and protection as provided in Japan, and to exempt any taxes or other fiscal levies which may be imposed in connection with the transfer of military technologies. (Paragraph 5)

Outline of Detailed Arrangements for the Transfer of Military Technologies

I. SCOPE

1. The Arrangements provide the general conditions and procedures applicable to the transfer to the DoD or a U.S. commercial entity of military technologies as defined in the Annex of the Exchange of Notes.

2. The military technology to be transferred, the persons who will be party to the transfer, and the detailed terms and conditions of the transfer shall be set forth in a separate memorandum concluded between the relevant Japanese authorities and the DoD for each transfer in accordance with the annexed format.

3. The implementation of the Arrangements and the separate memoranda will be consistent with the applicable laws and regulations of each country.

II. TECHNOLOGY TRANSFER

1. The JDA and/or the MITI shall take necessary measures to transfer the military technology to the DoD or to a U.S. commercial entity under the terms specified in the Arrangements and a separate memorandum for each transfer, and in accordance with direct contractual arrangements between the parties (except in the case of transfer from the JDA to the DoD). Such contractual arrangements shall be made subject to and governed by the terms of the Arrangements and the applicable separate memorandum.

2. All changes, modifications or improvements developed as a result of the use of the military technology transferred will be transferred to the JDA in the case of the transfer of JDA military technology or to a Japanese commercial entity in the case of the transfer of commercial military technology. The recipient will be granted a nonexclusive and irrevocable license to use such changes, modifications or improvements.

The JDA and the Japanese commercial entity, if they so choose, may waive the right to receive changes, modifications or improvements and may request financial compensation thereof.

III. AUTHORIZED USE AND RETRANSFER

1. The transfer of military technology and the use of such technology shall be consistent with the Charter of the United Nations.

2. The military technology transferred shall be used only for DoD military purposes that may be further specified, as necessary, in the separate memorandum for each technology and shall not be transferred to any person or organization not an agent of the recipient of the military technology nor any third-country government, person or organization without the prior written approval of the GOJ.

These provisions on authorized use and retransfer shall apply not only to the transferred military technology, but also to the changes, modifications or improvements thereof, and to any hardware or product produced essentially through an application of the transferred military technology.

3. The changes, modifications or improvements transferred to the JDA shall be used in accordance with conditions concerning the use and retransfer equivalent to those applicable to the DoD when it receives military technology.

When a recipient of the changes, modifications or improvements is a Japanese commercial entity, obligations of such entity to comply with conditions concerning retransfer equivalent to those specified in 2 will be determined by direct contractual arrangements.

IV. FINANCIAL MATTERS

The DoD shall pay to the JDA any appropriate research and development nonrecurring cost recoupment fee for the JDA military technology transferred to the DoD. Such recoupment fee shall be agreed upon by the JDA and the DoD before the transfer is effected.

V. SECURITY

Any classified information or materiel which may be transferred under the Arrangements shall be furnished in accordance with the MDA Agreement of and shall, in particular, be afforded protection pursuant to Article III thereof, paragraph 5 (1) of the Exchange of Notes, and applicable security laws, Executive Orders, directives and regulations.

VI. GENERAL PROVISIONS

1. Financial obligations or expenditures incurred by the DoD under the Arrangements or a separate memorandum shall be subject to the authorization and appropriation of funds.
2. The Parties will, upon the request of either of them, consult regarding any matter relating to the application of the Arrangements or a separate memorandum. In case of controversy that may arise under the terms of the Arrangements or a separate memorandum, such controversy will be submitted to the appropriate levels of each Government for consultation and final decision.
3. All communications and other documents may be submitted in either the English or Japanese language, and shall be accompanied by a translation into the other language, whenever technically feasible, at the cost of the recipient Party.
4. The Arrangements may be amended by agreement between duly authorized representatives of the Parties.

(Annex) Format of separate memorandum

1. PURPOSE
2. TECHNOLOGY TO BE TRANSFERRED
3. PARTICIPANTS TO THE TRANSFER
4. USE OF THE TRANSFERRED TECHNOLOGY
5. RECOUPMENT
6. SECURITY
7. GENERAL PROVISIONS

APPENDIX B

THE HIGH-TEMPERATURE COMPOSITES TECHNICAL ASSESSMENT TEAM (TAT)

Team Members, High-Temperature Composites TAT

Dr. Ben A. Wilcox, Team Leader, Assistant Director for Materials Sciences, Defense Advanced Research Projects Agency (DARPA), Arlington, Virginia.

Mr. Jamieson C. Allen, Director, Defense Technology Trade Programs, Mutual Defense Assistance Office, U.S. Embassy, Tokyo, Japan.

Mr. Charles F. Bersch, Science and Technology Division, Institute for Defense Analyses (IDA), Alexandria, Virginia.

Dr. Steven G. Fishman, Program Manager, Office of Naval Research (ONR), Arlington, Virginia.

Dr. Shiro Fujishiro, Associate Director, Air Force Office of Scientific Research--Far East (AFOSR-FE), Tokyo, Japan.

Dr. Allan P. Katz, Materials Research Engineer, Air Force Wright Research and Development Center (WRDC)--Materials Laboratory, Wright-Patterson Air Force Base (AFB), Ohio.

Dr. Ed Lenoe, Assistant Director, Army Research Office--Far East (ARO-FE), Tokyo, Japan.

Dr. Merrill L. Minges, Director, Nonmetallic Materials Division, Air Force Wright Aeronautical Laboratories (AFWAL)--Materials Laboratory, Wright-Patterson AFB, Ohio.

Dr. Fred Pettit, Assistant Director, Office of Naval Research--Far East (ONR-FE), Tokyo, Japan.

APPENDIX C

SCHEDULE OF TAT ACTIVITIES
30 JANUARY-14 FEBRUARY 1989

SCHEDULE OF TAT ACTIVITIES
30 JANUARY-14 FEBRUARY 1989

30 January 1989 (Monday)

0800-0930	Working breakfast at U.S. Embassy's Jefferson Room with American Chamber of Commerce-Japan (ACC-J) High-Technology Committee.
0930-1015	Meeting with Embassy's Science Council.
1100-1130	Courtesy call on Mr. Teruo Suzuki, Director General, Research and Development, Japan Defense Agency (JDA). --Dr. Wilcox and Mr. Bersch, accompanied by Mr. Allen and Mr. Aka.
1400-1430	Courtesy call on Mr. Shinichiro Ohta, Director, Aircraft and Ordnance Division, Machinery and Information Bureau, Ministry of International Trade and Industry (MITI). --Dr. Wilcox and Mr. Bersch, accompanied by Mr. Allen and Mr. Aka.
1540-1600	Courtesy call on Mr. Ryozo Tsutsui, Director General, Technical Research and Development Institute (TRDI), JDA. --Dr. Wilcox and Mr. Bersch, accompanied by Mr. Allen and Mr. Aka.

31 January 1989 (Tuesday)

1000-1630	Visit to Ube Industries' Ube Laboratories, Ube City in Yamaguchi Prefecture. --All TAT members, accompanied by Mr. Akira Ryuzaki, JDA; met by Mr. Kotoku or Mr. Ishikawa, Ube Industries.

1 February 1989 (Wednesday)

1100-1730	Visit to Kobe Steel, Kobe City in Hyogo Prefecture. --All TAT members, accompanied by Mr. Akira Ryuzaki, JDA; met by Mr. Yasahara, Kobe Steel.

2 February 1989 (Thursday)

0730-0830	TAT working/planning breakfast.
1100-1730	Visit to Shimadzu's Sanjo Works, Kyoto City. --All TAT members, accompanied by Mr. Akira Ryuzaki, JDA; met by Mr. Ishida, Shimadzu.

3 February 1989 (Friday)

0800-1000 TAT working/planning breakfast.

1030-1645 Visit to Toray Industries' Otsu Works, Otsu City in Shiga Prefecture.
--All TAT members, accompanied by Mr. Akira Ryuzaki, JDA;
met by Mr. Tanaka, Toray Industries

4 February 1989 (Saturday)

1800-2000 TAT working session on TAT report and Keidanren presentation.

6 February 1989 (Monday)

0700-0800 TAT working/planning breakfast.

1100-1730 Visit to Toyota Corporate Research and Development Laboratories (CRDL), Nagakute Town in Aichi Prefecture.
--All TAT members, accompanied by Mr. Motoi Satake, JDA.

7 February 1989 (Tuesday)

1100-1700 Visit to Ishikawajima-harima Heavy Industries (IHI) Toyosu Laboratories, Koto-ku in Tokyo area.
--All TAT members, accompanied by Mr. Shigeyoshi Hata, JDA.

8 February 1989 (Wednesday)

1100-1630 Visit to Mitsubishi Electric Corporation (MELCO), Sagami Works, Sagamihara City in Kanagawa Prefecture.
--All TAT members, accompanied by Mr. Shigeyoshi Hata, JDA.

1800-2000 Meeting with Keidanren officials, Mr. Kuno, MGEN Oshima (JASDF, retired), and MGEN Nishida (JGSDF, retired), to finalize details of Keidanren presentation.
--Dr. Wilcox, Mr. Bersch, Mr. Allen, and Mr. Aka.

9 February 1989 (Thursday)

1000-1600 Visit to Tonen General Laboratories, Iruma City in Saitama Prefecture.
--All TAT members, accompanied by Mr. Motoi Satake, JDA.

1830-2030 Tonen Reception, Maronouchi Hotel.

10 February 1989 (Friday)

1100-1630 Visit to Nippon Carbon's Yokohama Works, Yokohama City in Kanagawa Prefecture.
--All TAT members, accompanied by Mr. Motoi Satake, JDA.

13 February 1989 (Monday)

1200-1600 Visit to Tonen's Kawasaki Works, Kawasaki City.
--All TAT members, accompanied by Mr. Motoi Satake, JDA.

1830-2030 TAT reception at New Sanno Hotel.

14 February 1989 (Tuesday)

0800-0930 Working breakfast at U.S. Embassy's Jefferson Room with the American Chamber of Commerce-Japan (ACC-J) High-Technology Committee.

1430-1630 TAT presentation to Keidanren, Hotel New Otani.
--All TAT members, accompanied by Captain Dziedzic, Mr. Hastings, Mr. Allen, and Mr. Aka.

--Keidanren presentation chairman: Dr. Kanamori, Chairman of Defense Production Committee and Vice Chairman of Keidanren.

--Dr. Wilcox, briefing on "DARPA/DoD Activities in Composite Materials."

--Dr. Minges, overview of the National Aerospace Plane (NASP) program

--Dr. Katz, overview of the Integrated High-Performance Turbine Engine Technology (IHPTET) initiative

--Attendance of more than 100, including personnel from Keidanren's Advanced Technology Committee.

APPENDIX D

QUESTIONS FROM COMPOSITES TAT SUBMITTED TO JAPANESE INDUSTRY PRIOR TO VISIT

Questions from Composites TAT to Japanese Industry

This list of questions is intended to amplify the "areas of interest" and "details of interest" given in the statement of Purpose of the Technology Assessment Team on High Temperature Composite Materials.

Fibers

1. What high performance fibers are being produced, or will soon be produced? By what process? From what precursor materials? What is the composition? By what name is it known or marketed?

2. What is the maturity and production readiness of the process? Are the precursor materials readily available in ample supply? Is special production equipment needed? Is production equipment proven to be capable? Can production quantity be increased quickly?

3. What is the current market for these fibers? How much is being used? For what applications? Is much of the current production used in-house? Are new applications and new markets expected in the near future? If so, what?

4. What are the physical and mechanical properties of these production fibers? (Microstructure, thermal stability, CTE, oxidation resistance, thermal conductivity, electrical properties, etc.) How is quality and uniformity monitored or assured?

5. Is there more than one class or grade of fiber? Is there any special treatment or coating used for particular applications?

6. What are current fiber costs? Are they expected to decrease as production expands? Please explain.

7. What is the corporate commitment to high performance fibers? That is, what is the current level of effort - in people and facilities for current production and in developing and producing higher performance fibers? Has the company made a large investment in facilities and equipment? Is one needed?

8. Are there any current connections with U.S. industry - licensing or sales agreements? If so, with whom and how extensive? If not, would you desire such connections and in what general form - licenses, coproduction, sales?

9. What is your domestic and U.S. sales strategy? Are you eager for new markets or is production matched to company needs and commitments to other Japanese industry?

10. What motivated your investment in fiber production? Is it a natural fall-out from other products? Or did you have a need for fibers that was not available elsewhere? Were you stimulated by university work or in-house research or reports of work outside Japan? Are your interests predominantly in high-volume commercial markets or are you interested in high performance (usually low volume and high cost) markets too?

11. What are your expectations for the future in new or better fibers, new applications, larger markets in and outside Japan?

12. We are interested in brochures, annual reports, journal articles, videos or other forms of information.

Composites

1. What metal or ceramic matrix composites are being produced, or will soon be produced? By what process? From what constituent materials? By what name are they known or marketed?

2. What is the maturity and production readiness of the process? Are the constituent materials readily available in ample supply? Is special production equipment needed? Is production equipment proven to be capable? Can production quantity be increased quickly?

3. What is the current market? How much is being used? For what applications? Is much of the current production used in-house? Are new applications and new markets expected in the near future? If so, what?

4. What are the physical and mechanical properties? (Microstructure, thermal stability, CTE, oxidation resistance, thermal conductivity, electrical properties, etc.) How is quality and uniformity monitored or assured?

5. Is there more than one class or grade? Is there any special treatment or coating used for particular applications?

6. What are current costs? Are they expected to decrease as production expands? Please explain.

7. What is the corporate commitment? That is, what is the current level of effort - in people and facilities for current production and in developing and producing higher performance composites? Has the company made a large investment in facilities and equipment? Is one needed?

8. Are there any current connections with U.S. industry - licensing or sales agreements? If so, with whom and how extensive? If not, would you desire such connections and in what general form - licenses, coproduction, sales?

9. What is your domestic and U.S. sales strategy? Are you eager for new markets or is production matched to company needs and commitments to other Japanese industry?

10. What motivated your investment? Is it a natural fall-out from other products? Or did you have a need that was not available elsewhere? Were you stimulated by university work or in-house research or reports of work outside Japan? Are your interests predominantly in high-volume commercial markets or are you interested in high performance (usually low volume and high cost) markets too?

11. What are your expectations for the future in new or better composites, new applications, larger markets in and outside Japan?

12. We are interested in brochures, annual reports, journal articles, videos or other forms of information.

APPENDIX E

GUEST LIST FOR TAT RECEPTION

TAT RECEPTION AT THE NEW SANNO HOTEL

Guests:

TAT Members

Dr. Ben A. Wilcox
Dr. Allan P. Katz
Dr. Merrill L. Minges
Mr. Charles F. Bersch
Dr. Fred Findeis
Dr. Shiro Fujishiro
Dr. Ed Lenoe
Dr. Fred Pettit
Mr. Jamieson C. Allen

U.S. Embassy

(R) Dr. Richard Getzinger, Science Counselor
(R) Mr. William McPherson, Science Attache
(R) Mr. Alex DeAngelis, National Science Foundation Attache
(A) Captain Walter T. Dziedzic, Chief, MDO
(A) Mr. Norman S. Hastings, Deputy Chief, MDO

Japan Defense Agency (JDA)

(A) Mr. Masaji Yamamoto, Director General, Bureau of Equipment (BOE)
(A) Mr. Siro Nagato, Director, Coordination Division, BOE
(A) Mr. Shinshiro Yamazaki, Principal Deputy Director, Coordination Division, BOE
(A) Mr. Yoshifumi Fujita, Deputy Director, Coordination Div, BOE
(A) Mr. Hiroshi Marui, Deputy Director, Coordination Division, BOE
(A) Mr. Akira Ryuzaki, Deputy Director, Coordination Division, BOE
(A) Mr. Nobuhiro Beppu, Director, R&D Planning Division, BOE
(A) Mr. Shigeyoshi Hata, Deputy Director, R&D Planning Div, BOE
(A) Mr. Motoi Satake, Deputy Director, R&D Planning Div, BOE

(A) Mr. Teruo Suzuki, Director General, Research & Development

(A) Mr. Ryozo Tsutsui, Director General, Technical Research and Development Institute (TRDI)
(A) Dr. Sachio Uehara, Director, Plans & Programs Division, TRDI

Ministry of International Trade and Industry (MITI)

(R) Mr. Shinichiro Ohta, Director, Aircraft and Ordnance Division
(R) Mr. Masanori Suzuki, Deputy Director, Aircraft and Ordnance Div
(R) Mr. Yuji Nagahama, Deputy Director, Aircraft and Ordnance Div

KEIDANREN

(A) Mr. Hiroshi Morikawa, Secretary General, Defense Production Committee (DPC)
(A) Mr. Kokichi Morimoto, Deputy Secretary General, DPC
(A) Maj Gen (Ret) Naohiko Ohshima, Research Associate, DPC
(A) Maj Gen (Ret) Tsukasa Nishida, Research Associate, DPC
(A) Mr. Akihiko Kuno, Staff, DPC

Japanese Industries

(A) Mr. Koji Yamaguchi, Senior Managing Director, Corporate R&D, New Products Division, Ube Industries, Ltd.

(A) Dr. Toshio Nagasawa, R&D Senior Staff, Corporate R&D, New Products Division, Ube Industries, Ltd.

(A) Mr. Yamamura, Ube Industries, Ltd.

~~Mr. Keisaburo Ohwada, General Manager, Defense Equipment & Materials, Administration Dept, Kobe Steel, Ltd., Tokyo~~

(Substitute) Mr. Shigeo Suzuki, Manager, Defense Equipment & Materials, Administration Dept, Kobe Steel, Ltd., Tokyo

~~Mr. Katsuro Ito, General Manager, New Material Dept, New Business Development Div, Kobe Steel, Ltd., Kobe~~

(Substitute) Dr. Takao Mizoguchi, Kobe Steel, Ltd.

(A) Mr. Shin Izutsu, Managing Director, President, Aircraft Equipment & Industrial Machinery Group, Shimadzu Corp.

(A) Mr. Motoaki Funatsu, Director/General Manager, Aircraft Equipment Division, Shimadzu Corp.

(A) Mr. Toshio Toura, Director, Toray

(A) Mr. Junichi Matsui, General Manager, Advanced Composite Technology, Toray

(A) Mr. Ken Tanaka, General Manager, Carbon Fiber, Toray

(R) Mr. Osami Kamigaito, Director, Toyota Central Research Laboratory

(R) Mr. Toru Arai, Director, Toyota Central Research Laboratory

(A) Mr. Chiaki Aoki, Associate Director and Vice President, Technical Development, Ishikawajima-Harima Heavy Industries Co., Ltd. (IHI)

(A) Mr. Tohru Ishikawa, Associate Board Director and Vice President, Aero-Engine & Space Operations, IHI

(A) Mr. Tetsuo Tamama, Manager, Defense System-Technology Exchange, Electronics System Div, Mitsubishi Electric Corp.(MELCO)

~~Mr. Shunichi Mori, Manager, Marketing Development, Electronics System Div, MELCO~~

(Substitute) Mr. Minoru Usami, General Manager, Government Requirement Marketing Division, MELCO

~~Mr. Jun Inokuchi, Director, Towa Nenryo Kogyo K.K.~~

(Substitute) Mr. Iguchi, Towa Nenryo Kogyo K.K.

(A) Mr. Mitsuo Matsumura, General Manager, New Business Development Department, Towa Nenryo Kogyo K.K.

~~Dr. Haruo Teranishi, Deputy Director of Engineering & Development Division, Nippon Carbon Co., Ltd.~~

(Substitute) Mr. Yoshikazu Imai, Assistant Manager of Management Laboratories, Nippon Carbon Co., Ltd.

(A) Dr. Hiroshi Ichikawa, Deputy of Management Laboratories, Nippon Carbon Co., Ltd.

APPENDIX F

THERMAL STABILITY AND ENVIRONMENTAL EFFECTS

Frederick S. Pettit

CONTENTS

APPENDIX F
THERMAL STABILITY AND ENVIRONMENTAL EFFECTS

A. INTRODUCTION

Depending upon the application, fibers or composites with fibers may be subjected to elevated temperatures and different gas environments. It is therefore necessary to determine the effects of such conditions on the fibers' properties and to characterize any significant degradation processes. In comparing the thermal stabilities of fibers in different environments, there are certain factors which can be expected to significantly affect performance, and which therefore should be examined in an initial evaluation. Temperature and the gas environment are the two most critical parameters. The gas environmental effects should be compared at least for exposures in oxygen, in an inert gas, and in vacuum. In addition, in composites the reactions occurring between the fiber and the matrix at elevated temperatures should also be examined when possible. Another important consideration is the means by which the effects of exposure are measured. The most desirable procedures for fibers and composites are to examine reaction products and to measure mechanical properties such as yield strength or breaking strength as functions of time. In the following, the thermal stabilities of carbon and ceramic fibers are examined and compared by using data supplied by the manufacturers.

B. CARBON FIBERS

Currently carbon fibers are made from either PAN or pitch. In regard to thermal stability and environmental effects, there is no significant difference between PAN- and pitch-based fibers. In Fig. F-1 (Ref. 1) the oxidation rates of several types of pyrolytic graphite are presented as functions of temperature. These rates are high because gaseous products are formed. At temperatures below 600-800°C the rates are controlled by a chemical reaction involving the adsorption-dissociation-desorption of gases. At higher temperatures the rates are limited by the supply of oxygen. The temperature of the transition from the lower temperature controlling process to the controlling process at higher temperatures is determined by a number of factors, including the magnitudes of the

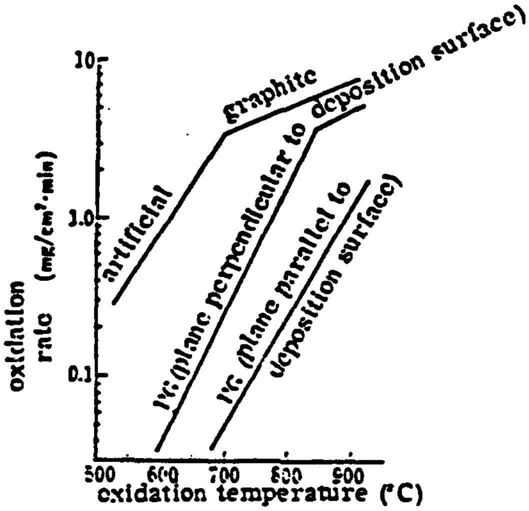

Figure F-1. Oxidation Rates of Pyrolytic Graphites (2300°C Deposition) in Air. (Source: Ref. 1)

rates of the lower temperature controlling process, the flow rates of the gas over the specimens, and the sizes of the specimens. At the higher temperatures there is no dependence of rate on the type of carbon being oxidized. At the lower temperatures, where the oxidation rates are controlled by the surface reaction, the rates can be affected by impurities in the carbon fibers, the degree of graphitization, and crystalline orientation (Fig. F-1). In applications involving short-term use, such as the space shuttle, rates of about 0.06 mg/cm^2-min (Ref. 2) may be acceptable compared to 0.06×10^{-2} mg/cm^2-min for long-term use, as in a gas turbine. Inspection of Fig. F-1 shows that acceptable rates for short-term exposures may be obtained at temperatures below 700°C; however, for long-term exposures in oxygen carbon cannot be used above 400°C regardless of its purity or structure. This is substantiated by the data presented in Fig. F-2 (Ref. 3) for several different carbon fibers.

Carbon is inert to gases such as helium, argon, and nitrogen, and it therefore can be used at extremely high temperatures (2500°C) in these environments as well as in high vacuum. The oxygen pressure in such environments must be low enough, however, that no significant quantities of carbon monoxide are formed.

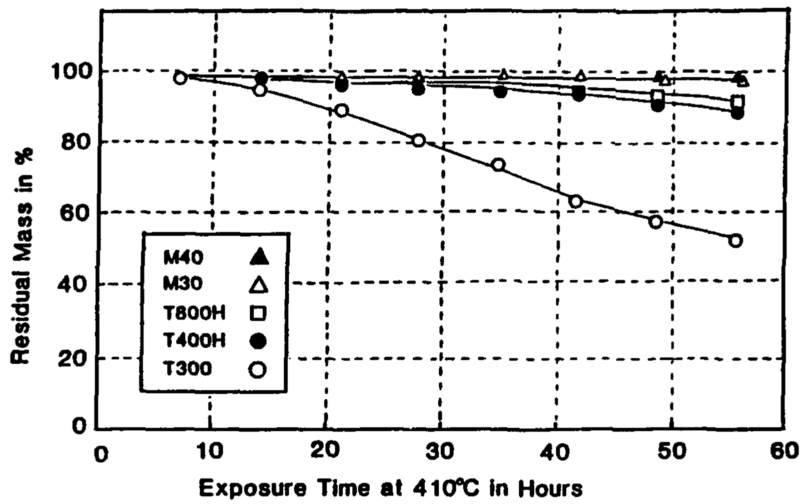

Figure F-2. Thermal Oxidation Stability of "Torayca" Carbon Fiber in Air. (Source: Ref. 3)

C. CERAMIC FIBERS

Before a discussion of thermal stability of the ceramic fibers, it is helpful to first examine the compositions of the available fibers. The approximate compositions of the fibers are as follows:

Company	Product	Approximate Composition
Ube Industries, Ltd.	Tyranno Fiber	$Si_{1-x}Ti_xC$ + oxygen (Typical titanium and oxygen concentrations are 2 and 18 weight percent, respectively. Ti can be as high as 7 percent.)
Nippon Carbon Co., Ltd. (NCK)	Nicalon	69.6 weight percent SiC + 20.8 percent SiO_2 + 9.6 percent free carbon (58.3 percent Si, 30.4 percent C, 11.1 percent O, 0.2 percent H)
Toa Nenryo Kogyo Co., Ltd. (Tonen)	Tonen Si_3N_4	Si_3N_4 + oxygen + carbon (1 weight percent oxygen, 0.5 weight percent carbon)
Shimadzu Corporation	Glass Fiber	Silicon oxynitride glass + Mg, Ca, Al (Up to 7 percent nitrogen; metal oxide permits more nitrogen to be added with no crystallization.)

Inspection of these compositions shows two important points with regard to thermal stability. First, all four compositions will react with oxygen to form SiO_2. Their resistance to oxidation attack therefore will be dependent upon the protectiveness of this reaction product, which will be influenced by impurities such as Mg and Ca. At very high temperatures (1400-1600°C) the formation of gases such as CO or nitrogen at pressures greater than 1 atmosphere may be possible. Secondly, in vacuum, or in environments with low oxygen pressures, volatile products of silicon monoxide may be formed. This will be especially so for the fibers with large fractions of SiO_2. In the following discussion, the thermal stability of each of the ceramic fibers will be reviewed.

The Tyranno fiber is reported to retain more than 95 percent of its strength after 6 hours exposure in nitrogen at 1300°C and after 6 hours exposure in air at 1000°C (Ref. 4). Such properties are consistent with this fiber's composition. It usually contains only 2 weight percent oxygen, and therefore formation of SiO in nitrogen should not be substantial. The fact that exposures in air cause more rapid deterioration of properties than in nitrogen shows that oxidation is occurring and that impurities may be affecting the protectiveness of the SiO_2 scale.

Tensile strengths of the Nicalon fiber after exposures at elevated temperatures in air and in argon are presented in Figs. F-3 and F-4, respectively (Ref. 5). The results are not substantially different from those for the Tyranno fiber. Adverse effects are observed earlier in air than in a less reactive gas, and the higher the temperature the shorter the time before such effects are observed. The composition of the Nicalon fiber is such that SiO_2 formation in air and loss of SiO in an inert gas are to be expected. While SiO_2 formation in air has been documented (Ref. 5) at temperatures above 1200°C, it has been proposed (Ref.5) that crystallite growth in Nicalon, carbon monoxide evolution during exposure in argon, and cracks in the SiO_2 scale are conditions that result in the decreased tensile strengths.

In Fig. F-5 (Ref. 6) the effects of oxygen on the retention of tensile strengths are presented for the "Tonen Si_3N_4" fiber. Effects become evident after less than 1 hour at 1200°C, which is similar to the results presented for the other fibers. It was reported (unpublished data) that the thermal stability of the Tonen fiber in nitrogen could be improved by changing its composition. Data were presented showing tensile strength retention at 1300 and 1400°C after 1 hour exposures in nitrogen. Thermogravimetric data

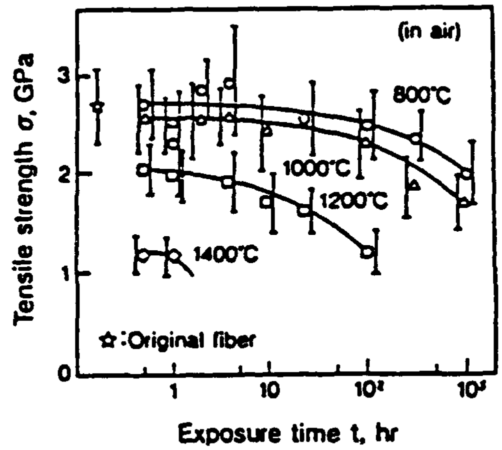

Figure F-3. Tensile Strength of SIC Fibers After Exposure in Air. (Source: Ref. 5)

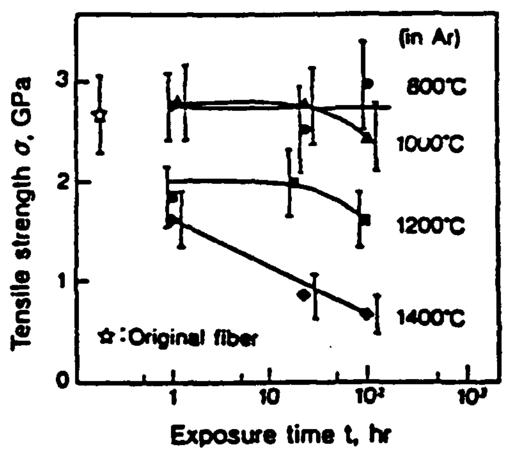

Figure F-4. Tensile Strength of SIC Fibers After Exposure in Argon Gas. (Source: Ref. 5)

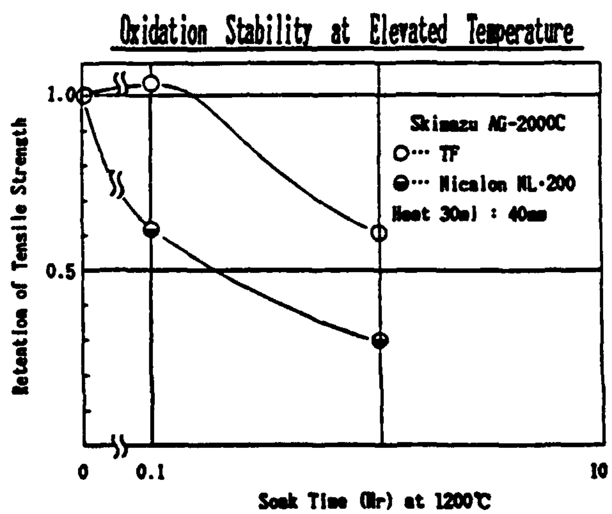

Figure F-5. Oxidation Stability of Tonen Fiber and Nicalon in Air. (Source: Ref. 6)

were also presented for isochronal exposure (10°C/min) of this fiber at temperatures up to 1500°C in nitrogen and in air. A weight increase of about 5 percent was observed in air with substantially less in nitrogen.

The Shimadzu fiber cannot be used in air at temperatures above about 700°C, since it begins to oxidize. Evidently the protectiveness of the SiO_2 must be influenced by the Mg and Ca in this fiber. This fiber also was reported to have a significant reduction in tensile strength after exposure at 800°C in an inert gas environment.

D. COMPOSITES

Very scant data are available in regard to the effects of environments on composites or to describe matrix-fiber reactions. It was reported that carbon-fiber-reinforced aluminum alloys could not be used above 200°C, whereas use temperatures as high as 400°C were possible for SiC fiber as a reinforcement. Such proposed application temperatures indicate that reactions between the SiC fibers and aluminum alloy matrices are much less extensive than those with carbon fibers.

Data in Fig. F-6 (Ref. 7) on the composite Nicaloceram (Nicalon fiber in SiC matrix) showed that there was no decrease in flexure strength after 30 hours exposure in air at 1000°C. It was also reported that a similar composite (CERASEP made by SEP by a CVI process) has been exposed for 500 hours in air at 1100°C with no observed decrease in strength. No data were provided to show the amounts and types of reactions that occur when Nicaloceram is exposed at temperatures between 1000-1200°C. At these high temperatures some SiO_2 will be formed, and if it is not highly protective, some effects of the environment on the properties of this composite can be expected to occur eventually.

It was also proposed that the processes to prepare Nicalon can, with some modification, be used to apply coatings, Nicalocoat, to protect various materials, including carbon-carbon composites. Such coatings should be examined in more detail. Factors such as adherence to the substrate, the effect of thermally induced stresses, and maximum use temperatures must be evaluated.

E. SUMMARY

Carbon fiber and carbon-carbon composites cannot be used above 400°C in oxidizing environments unless coated with some material such as SiC to provide oxidation resistance. Furthermore, such coatings must usually also contain a component to seal cracks which develop in the coatings during thermal cycling.

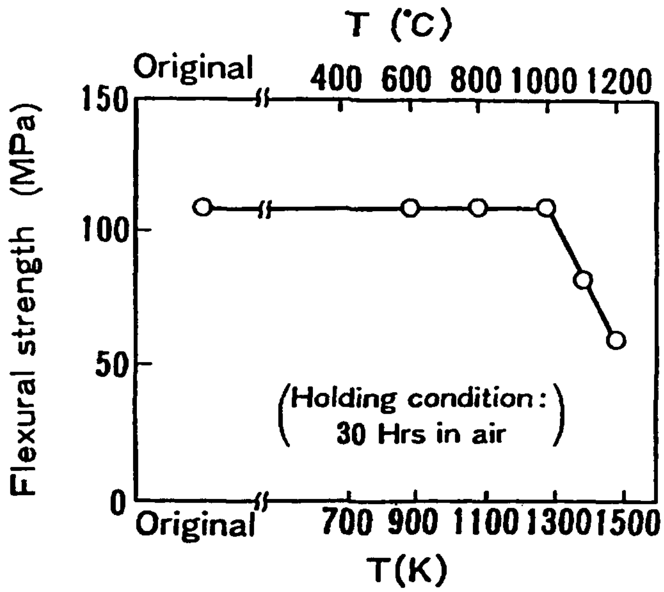

Figure F-6. Flexural Strength of Nicaloceram After Exposure for 30 hr in Air at Different Temperatures. (Source: Ref. 7)

Ceramic fibers such as the Tyranno and Nicalon fibers represent a very substantial improvement in oxidation resistance compared to carbon fibers. Nevertheless, these fibers do oxidize and impurities can have a very significant effect upon their performance. Depending upon the requirements that the fiber must satisfy, temperatures as high as 1000-1200°C in air may be possible.

In inert gases, or in nonoxidizing environments, all the fibers, including carbon, have extended high-temperature lives compared to those in oxidizing environments, and temperatures as high as 1300-1400°C are possible for ceramic fibers. Under these former conditions lives may be limited by crystallization, crystal growth, or vaporization of SiO. Carbon fibers may be used at temperatures in excess of 2000°C in inert environments.

The composite Nicaloceram possibly may be used for extended periods of time at temperatures as high as 1200°C. It is important to emphasize that, while this material has very impressive resistance to oxidation, it must rely upon the formation of a protective scale of SiO_2 for this oxidation resistance. The protectiveness of SiO_2 is affected by impurities. Sources of impurities are the composite and the environment. In some environments small amounts of impurities such as Na can be present, and they may cause the protectiveness of SiO_2 to be very significantly decreased.

In Fig. F-7 (Ref. 7) a schematic diagram to show possible use temperatures of Nippon Carbon products is presented. The environments have not been defined, nor have the useful lives at such temperatures been specified. The carbon-carbon composite must, of course, be coated when used under oxidizing conditions. Nevertheless, while the upper temperature limits would be reduced in air or oxygen, this figure presents a reasonable comparison of the possible application temperatures for some of the systems that have been discussed. A new MITI program has an objective to develop a fiber for use in air at 1700°C for short exposure times. At such temperatures SiO_2 may not be suitable as a protective barrier, and therefore totally different fibers will have to be considered.

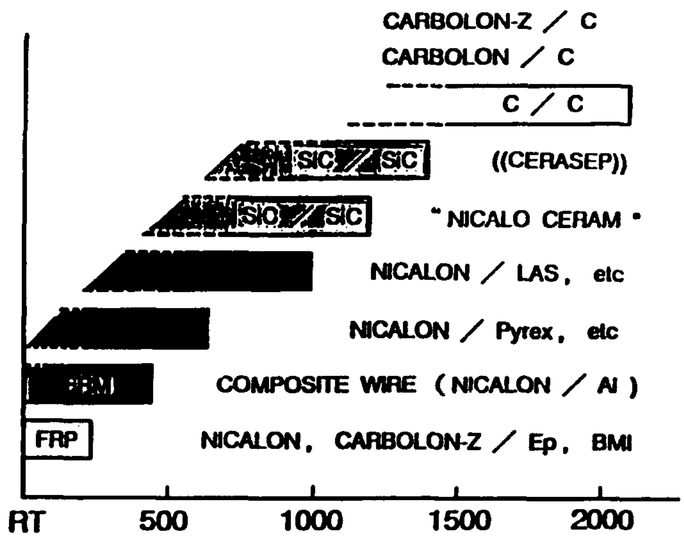

Figure F-7. Schematic Diagram Illustrating Possible Temperature Ranges of Application of Some of Nippon Carbon's Products. (Source: Ref. 7)

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